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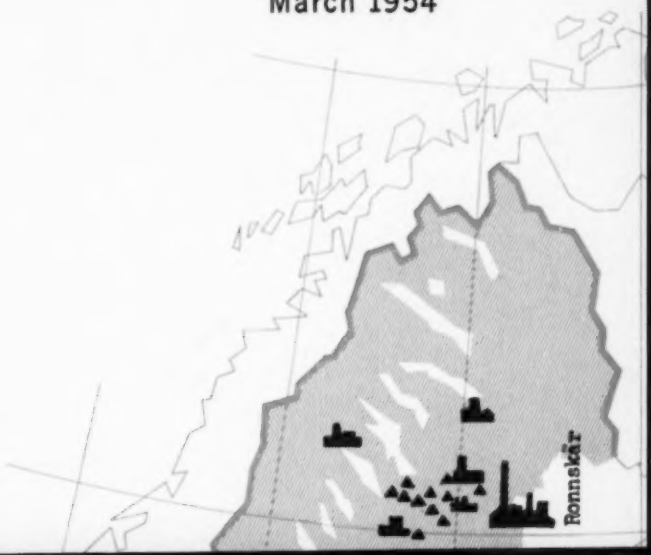
March 1954



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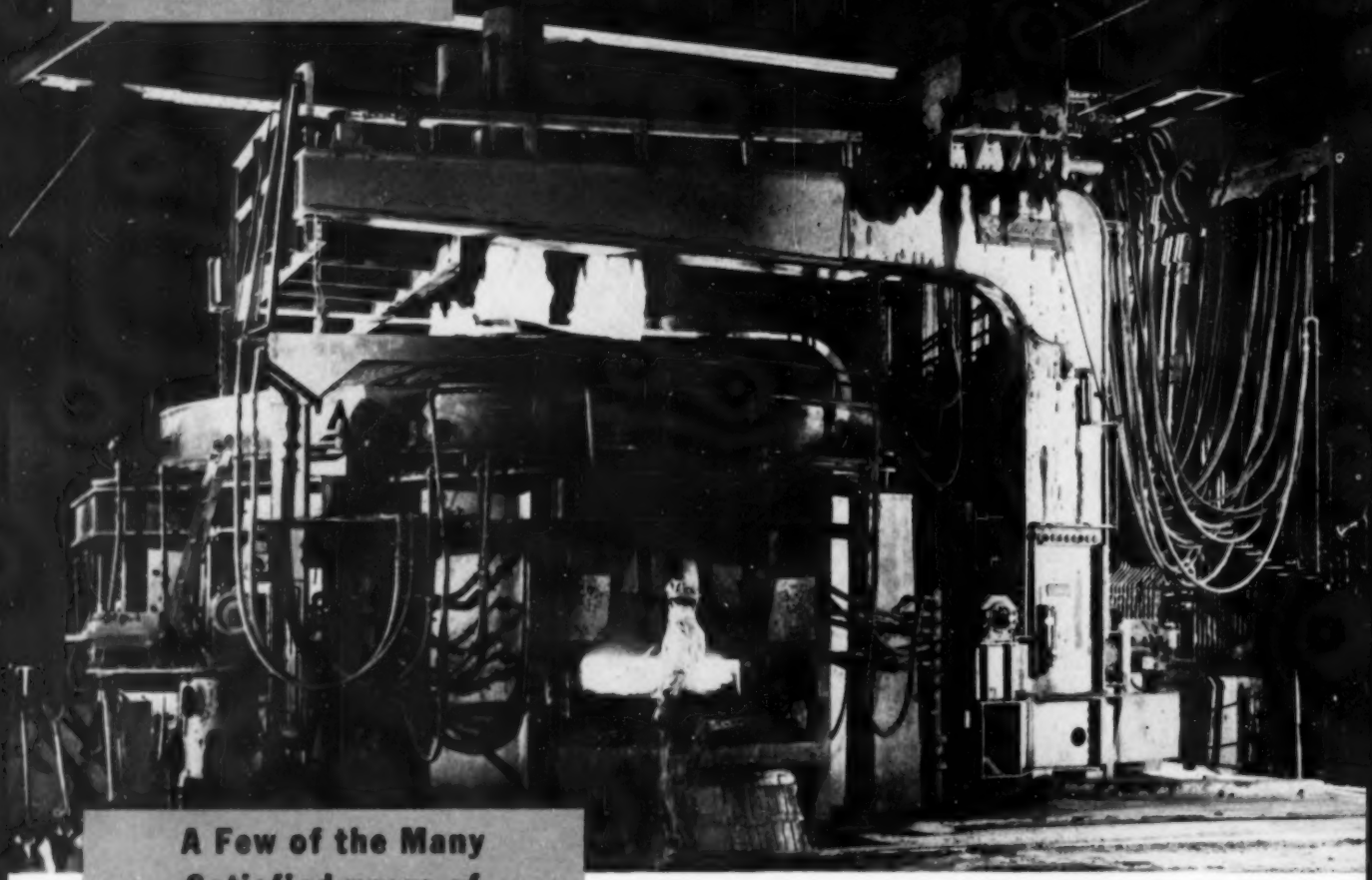

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COVER

The cover, designed by Harry Brocke, illustrates the story of copper smelting at the Boliden Mining Co., Sweden, which starts on p. 330. The Ronnskar smelter works, shown with its 145-meter high stack, is located at the mouth of the Skelleftea River and employs about 150 persons in its research laboratory.

NEWS

Meet the Authors	315	Journal of Metals Reporter	327
Employment Service	316	Drift of Things	328
Books for Engineers	317	AIME News	383
Industrial Notes	318	Coming Events	385
New Products	321	Personals	386
Trends	324	Obituaries	387

Blast Furnace, Coke Oven and Raw Materials Conference Program 381
National Open Hearth Steel Conference Program 382
New England Regional Meeting 382

TECHNICAL FEATURES

Copper Smelting in Boliden's Ronnskar Works Described — O. Herneryd, O. A. Sundstrom, A. Norro 330

Current Refractory Practice as Applied in Copper Smelting — W. F. Rochow, L. A. McGill 338

Direct Reading Spectrograph Gives Accurate Control in Aluminum Production — D. L. Colwell, O. Tichy 343

Phases in Titanium Alloys Identified by Cumulative Etching — E. Ence, H. Margolin 346

TRANSACTIONS

Application of the ARL Quantometer to Production Control in a Steel Mill — H. C. Brown 349

Distribution of Manganese Between Slag and Metal Under Reducing Conditions — J. E. Stukel, J. Cacubinsky 353

Copper Converting Practice at American Smelting & Refining Co. Plants — F. W. Archibald 358

A Quantitative Measure of Temper Embrittlement — N. Brown 361

Constitution and Mechanical Properties of Titanium-Hydrogen Alloys — G. A. Lenning, C. M. Craighead, R. I. Jaffee 367

Load-Temperature History of Lattice Strain in Aluminum Alloy — D. Rosenthal, M. Kaufman 377

TECHNICAL NOTES

Occurrence of Silicon Carbide in the Fe-C-Si System — J. C. Fulton, J. Chipman 356

On the Effects of Oxygen on Molybdenum — R. E. Maringer, A. D. Schwope 365

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ELECTROMET Data Sheet

A Digest of the Production, Properties, and Uses of Steels and Other Metals

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COLUMBIUM and TANTALUM

Strategic Combination for Imparting Strength and Stability to High-Temperature Metals

Gas turbines for jet-aircraft engines, and for other similar engines being developed for marine and railway transportation, have greatly increased the demand for high-temperature metals. The present most satisfactory metals are either iron-base, nickel-base, or cobalt-base. They are used in the form of castings and hot-worked products, such as forgings, bars, and sheets.

Many of these special metals contain columbium because of its beneficial effect on high-temperature strength in both cast and wrought products. Investigations have shown that columbium is one of the key alloys for imparting high-temperature strength and stability in metals suitable for operating temperatures up to 1500 deg. F. and above.

Need for New Alloy

Originally, a ferrocolumbium alloy containing approximately 55 per cent columbium and 5 per cent tantalum was employed in the production of many of these high-temperature metals. However, with the increased use of high-temperature metals and columbium-bearing 18-8 stainless steels, it became necessary to utilize the tantalum-rich columbium ores.

ELECTROMET's Research Laboratories investigated whether an alloy containing more tantalum and less columbium would be equally satisfactory for producing the high-temperature metals. Columbium and tantalum alone, as well as combination alloys of columbium plus tantalum, were tested.

Results of Tests

The alloy N-155 was selected for tests. It is an iron-base alloy with the following approximate analysis:

Chromium	20 per cent
Nickel	20 per cent
Cobalt	20 per cent
Tungsten	3 per cent
Molybdenum	2 per cent
Columbium	1 per cent
Nitrogen	0.15 per cent
Carbon	max. 0.35 per cent

The data in the table below describe the mechanical properties, at room temperature, of this low-carbon N-155 alloy modified with columbium and tantalum alone, and with combinations of columbium plus tantalum. The metal modified with columbium has good strength and high ductility at room temperature. These same good properties are obtained when tantalum, or tantalum plus columbium, is substituted for the columbium.

Stress-to-rupture tests were also conducted on these same modified low-carbon N-155 alloys at 1350 and 1500 deg. F. The data show (see table) that when the columbium is replaced with a mixture of columbium plus tantalum, the strength of the metal remains substantially unaffected at 1350 and 1500 deg. F. Also, when all of the columbium is replaced with tantalum, the strength of the metal at 1500 deg. F. is equivalent to that obtained with columbium. Hence, from the standpoint of high-temperature strength, columbium and tantalum can be used interchangeably, or in combination.

Help to Industry

ELECTROMET has developed an alloy containing approximately 20 per cent tantalum and 40 per cent columbium for use in high-temperature metals and stainless steels. It is known as ELECTROMET ferrotantalum-columbium.

Industrial experience with this alloy has confirmed the favorable results of the experimental work. The alloy has already aided considerably in augmenting the supply of columbium alloys, since it is just as effective as ELECTROMET ferrocolumbium, with 50 to 60 per cent columbium, for giving strength at high temperatures.

It should be added to a thoroughly deoxidized metal bath to obtain the best results. A recovery of about 90 per cent for the columbium and 80 per cent for the tantalum may be expected.

For further information regarding alloys for high-temperature metals, write to the nearest ELECTROMET office: in Birmingham, Chicago, Cleveland, Detroit, Houston, Los Angeles, New York, Pittsburgh, or San Francisco. In Canada: Welland, Ontario.

Properties of Modified N-155 Alloys Compared*

Typical Analysis, %**	With Columbium	With Columbium and Tantalum		With Tantalum
Columbium	1.13	0.58	0.49	....
Tantalum	0.08	0.64	0.33	1.47
Carbon	0.12	0.13	0.13	0.12
Nitrogen	0.13	0.14	0.14	0.14
At Room Temperature				
Tensile Strength, psi	119,000	123,200	117,500	122,100
Yield Strength, psi	56,700	59,500	52,000	59,100
Elongation in 2 in., %	52	47	57	54
Reduction of Area, %	69	65	69	56
At 1350 deg. F.				
Stress to Cause Rupture, psi				
In 100 hr.	31,000	34,000	33,500	....
In 1000 hr.	23,000	25,000	23,000	....
At 1500 deg. F.				
Stress to Cause Rupture, psi				
In 100 hr.	20,000	20,000	20,500	20,000
In 1000 hr.	15,000	14,000	15,500	15,000

*Tests made on standard samples from one-inch round bars, water-quenched from 2250 deg. Fahrenheit.
**Composition of the base alloy is given in the text.

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Meet The Authors

W. F. Rochow (p. 338) who is vice-president of research and technical sales, Harbison-Walker Refractories Co., has been with the company since 1912. His early years with the company were engaged in research and then technical sales. Mr. Rochow was named assistant general sales manager, and later assistant to president. Born in Columbia, Pa., he is a graduate of Pennsylvania State University, (B.S., 1912). Mr. Rochow is a Member of the Metals Branch, AIME, and also holds membership in the American Ceramic Society. He previously had a paper published in JOURNAL OF METALS October 1951 on new developments in metallurgical refractories.

W. F. ROCHOW

O. HERNERYD

O. Herneryd (p. 330) received his chemical engineering degree from the Royal Institute of Technology in 1930. He is presently general manager of the smelter Roonskars works, Boliden Mining Co. He is an AIME Member and also holds membership in the ACS.

D. L. Colwell (p. 343) is a Member of the Cleveland AIME Section, as well as professional societies here and abroad. Mr. Colwell graduated from the University of Chicago in 1916. He is now director of laboratories, Apex Smelting Co., Chicago. Mr. Colwell has been with Apex for approximately eight years. He had been associated with Stewart-Warner, die casting div. for 20 years. Mr. Colwell has been engaged by the government as a consultant at various times. He spent five years in Washington and Japan as a nonferrous consultant and in 1949 was on the aluminum team sent to Europe. He has addressed several local sections on die casting.

R. E. Maringer (p. 365) graduated from the University of Detroit in 1950, (B.S.). He has been with Battelle Memorial Institute since January 1951 in the physical metallurgy dept. He previously presented a Research in Progress note before the AIME Annual Meeting in 1952. His main interests outside of his work are photography and astronomy.

H. C. Brown (p. 349) has been with Armco Steel Corp. since 1942, with the exception of 2½ years, when he served in the Navy. He started as an analyst in the alloy laboratory, east works div., Middletown. In May 1947 he was transferred to the research laboratories as an analyst in the chemical laboratory. Two years later he joined the spectrographic laboratory. Armco transferred him to Butler, Pa., in 1951 to the new laboratory that was being installed. Mr. Brown was made supervisor of the chemical and spectrographic laboratory in July 1953. Born at Logan, Ohio, he received his B.S. from Ohio University. His hobbies are sports and gardening.

O. Tichy (p. 343) is chief metallurgist for the Apex Smelting Co., Chicago. Born in Cleveland, he holds A.B. and M.S. degrees. Mr. Tichy joined the National Smelting Co. in 1933 as a chemist. He was promoted to metallurgist in 1937 and named assistant to laboratory director in 1945. He joined the Apex Smelting Co. in 1948 as chief metallurgist. In his free time, Mr. Tichy enjoys photography, gardening, and is a sports manager. He holds membership in AIME, AFS, ASTM, and ASM.

Elmars Ence (p. 346) is a native of Riga, Latvia. He graduated from the University of Latvia in 1934 and was an assistant professor at the University for ten years. In 1944 he joined the Heinkel Aircraft Works in Germany and from 1946 to 1949 was lecturer of analytical chemistry at the Baltic University, Hamburg. Since 1951 Mr. Ence has been a research associate at New York University. His research work includes X-ray crystallography and stress corrosion. When not engaged in these activities, he enjoys painting, cartooning, and sailing.

James C. Fulton (p. 356) is a member of the New York City AIME Local Section. He holds a B.S. from the University of Pennsylvania and S.M. and Sc.D. from Massachusetts Institute of Technology. Dr. Fulton is presently a research metallurgist at Allegheny Ludlum Steel Corp., Watervliet, N. Y. An AIME Member, he has had papers published in 1951 and 1953. In his free time, Dr. Fulton turns his hand to carpentry. He also enjoys swimming, playing tennis, and reading.

M. Kaufman (p. 377) is a graduate of City College of New York, and University of California at Los Angeles, where he is presently an instructor in engineering. He enjoys good music and plays the piano in addition to collecting records. Mr. Kaufman has been an engineer in the Merchant Marine and for three years was an engineering design draftsman for the Douglas Aircraft Co. Since 1947 he has held various positions at UCLA.

Allan Norro (p. 330) joined the Boliden Mining Co. in 1941, after gaining experience at various metallurgical operations in Sweden and Germany. He was named assistant smelter superintendent in 1945 and has been superintendent of the copper smelter since 1948. Mr. Norro was awarded the American Scandinavian Foundation Fellowship for 1948 to 1949 for post graduate studies at the University of Utah and different smelters in the United States and Canada. He is a graduate metallurgical engineer from the Royal Institute of Technology, Stockholm. He is a member of the Svenska Teknologforeningen. As his hobby, he enjoys sailing.

ALLAN NORRO

O. A. SUNDSTROM

O. A. Sundstrom (p. 330) is general superintendent of the copper works at Boliden Mining Co., Skelleftehamn, Sweden. He is a graduate of the Royal Institute of Technology, Sweden and holds a chemical engineering degree.

F. W. Archibald (p. 358) joined the American Smelting & Refining Co., El Paso, after his graduation (B.S) from the Montana School of Mines in 1940. In 1943 he was transferred to Garfield, Utah as metallurgist and in March 1953, Mr. Archibald was named assistant superintendent of the El Paso smelting works. From 1944 to 1946 he was on active duty overseas as an officer in the Naval Reserves. A Member of the AIME El Paso Section, his main avocation is golf.

Lincoln A. McGill (p. 338) has been associated with Harbison-Walker Refractories Co. for 24 years. He is presently district sales manager located in San Francisco. Mr. McGill was born at Stockton, Calif. Before joining Harbison-Walker he had been a salesman in various territories throughout the west. *Reaction Temperatures Between Refractories* is the title of a paper he presented before the American Ceramic Society. This AIME Member enjoys golf and gardening as his spare time interests.

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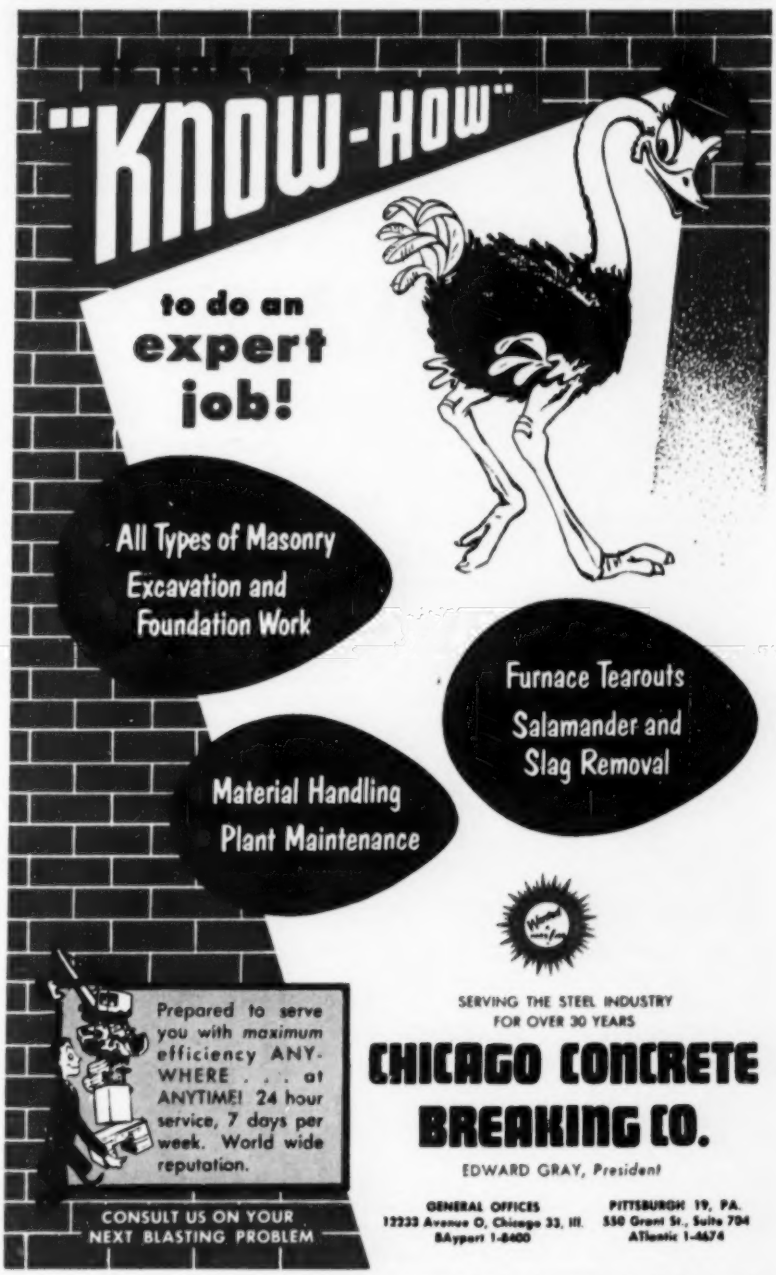
Materials and Processes, second edition, by James F. Young. *John Wiley & Sons, Inc.* $8.50, 1074 pp., 1954. The second edition has been expanded by 50 pct and brought completely up-to-date. It includes a thorough coverage of the developments of the last ten years. The book provides all the interrelated information needed to apply the engineering fundamentals of materials and processes to the design, production, and control of products. Recent advances in physical metallurgy and chemistry, useful material property data and process tolerances, latest industry practices, inclusive summary of topical literature, and approximately 500 illustrations are included. The book is one of a series written in the interest of the General Electric Engineering Educational Programs.

Heat-Resisting Steels and Alloys, by C. G. Conway. *D. Van Nostrand Co., Inc.* $5.00, 160 pp., 1953. Data has been included in this book covering the majority of steels and alloys produced commercially in the United States and United Kingdom. For easy reference, the materials have been divided into various classes and data for each is arranged under the following headings: Typical composition; common uses; advantages and disadvantages; creep data, both graphical and tabular; and physical and mechanical properties. For engineers and designers who require adequate information on which to base the design of components used in construction of gas turbines, furnaces, power station plants, etc., the book provides a convenient source of reference.

Report on the Elevated-Temperature Properties of Chromium-Molybdenum Steels. American Society for Testing Materials. $4.75, 212 pp., 1953. This is the second in a current series of reports prepared under the auspices of the data and publications panel of the ASTM-ASME joint committee on effect of temperature on the properties of metals. Summary curves for 23 steels, 200 figures, and more than 250 data sheets for 52 steels are included. The appendix contains the primary data from which the summary curves were prepared. The data sheets include the chemical composition, processing data, heat treatment, and other pertinent information about the steels included in the survey.

Das Blech Und Seine Prufung, by Gerhard Oehler. *Springer-Verlag.* Approximately $7.00, 297 pp., 1953.—This is a comprehensive treatment of the working and testing of steel and nonferrous sheet metal. It is intended for the use of works managers, plant engineers, and foremen. The book has five main divisions: Sheet and strip for pressworking and drawing; workability of various sheet metals; sheet metal thickness; strength testing; other tests—including chemical, surface, finish, and metallographic.

Radioactivity and Radioactive Substances, fourth edition, by Sir James Chadwick and revised and supplemented by J. Rotblat. *Pitman Publishing Corp.* $3.00, 120 pp., 1953.—A book on classical radioactivity, it is intended to serve as a primer in this field or as general introduction to nuclear physics. Simple treatment is employed in discussion of basic phenomena of natural radioactivity, and how these have led to present concepts of the structure of matter. A chapter on nuclear structure has been added to this edition.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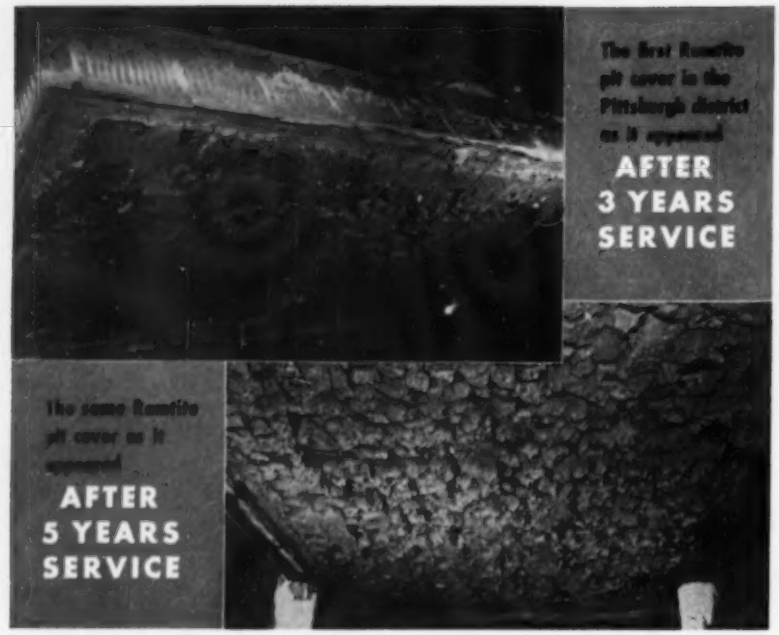
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Industrial Notes . . .

• Zirconium oxide production of the new plant of the Zirconium Corp. of America, at Solon, Ohio is expected to reach 8000 lb per day when full capacity is reached later this year. A new process takes four to five hr from raw material to finished product, according to George R. Sylvester, company president. Price range of the Zirconium Corp. product will be from $1200 to $1700 per ton, compared with $900 to $3000 per ton for comparable competitive grades.

• Youngstown Sheet & Tube Co.'s Brier Hill plant is overhauling what is said to be the oldest, fastest, and most powerful blooming mill steam engine in the world. The engine, which once powered the 40-in. blooming mill being torn down, will be used for the 44-in. blooming mill now being installed. The engine was built in 1913 by the William Tod Co., now part of United Engineering & Foundry Co. The engine develops 25,000 hp at top speed of 106 rpm. It operates at an average speed of 90 rpm. It has a high pressure cylinder, 44 in. in diam, and a 72-in. low pressure cylinder.

• General Services Administration announced an agreement with E. I. duPont de Nemours & Co., of Wilmington, Del., under which the company will begin design and engineering studies for the erection of a plant to produce 7000 to 8000 tons of titanium annually. Du Pont will spend a maximum of $600,000 of its own in studies and drawings preparatory to possible construction of a plant costing between $30 and $35 million. Edmund F. Mansure, General Services Administrator, said, "While there is no agreement that the plant will be built, the data are needed so that we will be able to conclude negotiations with the company for the additional production."

• U. S. iron and steel companies plan to invest about $775 million for expansion and improvement, bringing the nation's postwar investment to almost $6.4 billion. Iron and steel firms spent more than $1 billion in 1954. Medium and small companies have been sharing the expansion with the larger firms.

• General Electric engineers have successfully used tungsten in a commercial precision resistance thermometer. Advantages claimed for tungsten used as the sensing element of a resistance thermometer are high melting point and large change in resistance with temperature.

• Koppers Co., Inc., announced contruction of a new mechanical development laboratory for refinement and adaptation of present products and development of new products for the firm's metal products div. The new laboratory will be at Koppers South Baltimore plant. Housing will be in a one story building, 98x160 ft.

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• *Reynolds Metals Co.'s new Robert P. Patterson aluminum reduction plant, near Arkadelphia, Ark., is in operation. The plant has an annual rated capacity of 110 million lb of aluminum and cost an estimated $34 million. Reynolds' subsidiary, Reynolds Reduction Co., has substantially completed the La Quinta, Texas alumina plant, and is operating the installation. Rated output is 1000 tons of alumina per day.*

• Three steel industry foundations have established fellowships at Illinois Institute of Technology, Chicago, for training in mechanical and metallurgical engineering. The La-Salle Steel Foundation fellowships cover both mechanical and metallurgical engineering. Allegheny Ludlum Steel Corp. and the Signode Foundation are sponsoring one award each in metallurgical engineering. Applications for the fellowships must be made to the Office of Admissions by March 1.

• **Alloy Precision Castings Co., working with Mercast Corp., licensor of the frozen mercury pattern Mercast process, recently poured in the laboratory a titanium aircraft latch using a modified Mercast ceramic mold. Experiments are continuing and results are said to be "encouraging." However, Alloy Precision Castings states that pouring of titanium mercastings in production quantities will be limited by present nonavailability of satisfactory melting equipment and procedures.**

• International Nickel Co. of Canada, Ltd., has made the first shipment of metallic nickel under a contract calling for quick delivery of 120 million lb of the metal to the U. S. Government over a five year period ending in 1958. Shipment was from Inco's Port Colborne refinery. Deliveries will be made at a monthly rate of 2 million lb until the contract is filled.

• *Sixteenth session of the semi-annual X-ray Diffraction School sponsored by the North American Philips Co., Inc., for registrants who can come to New York City will be held at the company plant, 750 South Fulton Ave., Mount Vernon, N. Y. from April 19 to 23. Registration for the first four days will be limited to 125 and to 150 on the fifth day.*

• Metal Hydrides, Inc., Beverly, Mass., recently announced a new metallurgical development dept. The department will be headed by Emanuel Gordon, Metal Hydrides chief metallurgical engineer.

• Entire manufacturing facilities of Seaboard Refractories Co., Raritan Township, N. J., will be devoted to producing special refractories for Babcock & Wilcox Co., of New York. Under the agreement, Seaboard will discontinue its own sales activities and sales will be handled by B&W.

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New Products

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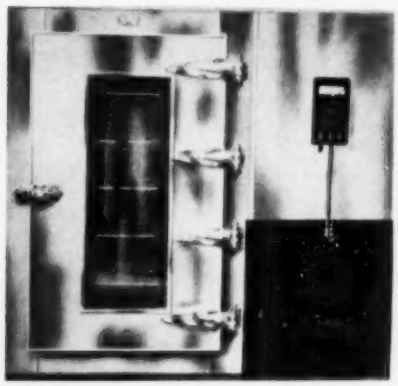
Students are Requested to Apply Directly to Manufacturers for Bulletins and Information

1—TEST CHAMBER: Model A-30-40FH, designed for large capacity testing at high and low temperatures, is available from *Webber Mfg. Co., Inc.* Temperature range is to −40°F on refrigeration cycle and to +200°F on heat cycle. Features include visible type controller, pat-



ented heat exchangers and multi paned visual port and apertures for connections to inner chamber.

2—NOZZLE HEATER: Application of a Termaband nozzle electric heater gives close temperature control. Results obtained are: Elimination of the freezing of die casting material in the nozzle at the end of the casting cycle, reduction of the tendency of the nozzle heater to overheat the die, and elimination of uncontrolled nozzle warping. *Thermal, Inc.*

3—CONTROL SYSTEMS: Complete control packages, designed for specific furnace, oven, kiln, or dryer applications are available from *Bristol Co.*

4—STRIP STRAIGHTENER: Machine has been developed for straightening thin strip from a few thousandths in thickness up to full capacity of the 20 ton self-contained hydraulic circuit, and as wide as 20 in. It is arranged to take strip from the coil, straighten it, and recoil it. *Medart Co.*

5—SPECTROSHELL MOLDS: *Baird Associates* offer new spectroshell molds for the production of sample pins for better emission spectrographic analysis. Single unit spectroshell molds measure 2x4x3½ in. overall and afford capacity for four pins each ¼x2¼ in.

6—ACETOGEN GAS: Especially suitable for brazing and silver soldering operations because the characteristics of the flame enable uniform heating of large areas of metal with no tendency for melting, burning, or change of temper, this gas is available from *Acetogen Cutting Gas Co., Inc.* One cylinder is equivalent to four cylinders of acetylene. The gas burns with a nonincandescent flame, is nontoxic and produces no soot or carbon deposits.

7—TEMPERATURE CONTROLLER: A new two-zone electronic temperature controller has been announced by the *Robertshaw-Fulton Controls Co.* The instrument uses a resistance temperature detector element as a sensing device in a bridge circuit in conjunction with a high gain phase sensitive amplifier relay unit. Standard ranges are 0 to 100, 300, or 500°F and C. Any range between the limits of −200° to +500°C can be supplied in conjunction with an appropriate resistance bulb from the Fielden list.

8—POLISHING DESK: Polishing desk with storage cabinets approximately 60 in. long, 27 in. deep, and 30 in. high, including two 12 in. swing spouts, 8 in. diam wash bowl has been introduced by *Buehler Ltd.*

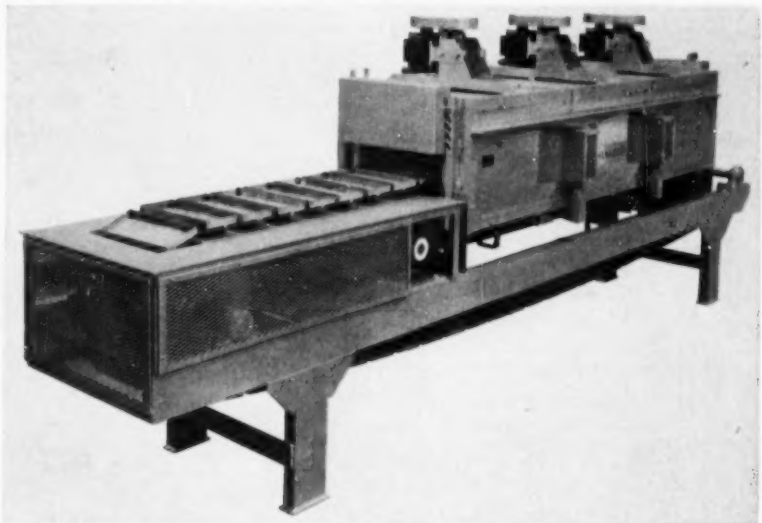
9—ELECTROPLATING RECTIFIERS: Rectifiers available in five basic models have been developed by *Wagner Brothers, Inc.* They are conservatively rated, 3-phase circuit, selenium type rectifier stacks connected for 5 pct ripple.

10—STEEL BAR: A free machining, open hearth, leaded steel bar is now available from *LaSalle Steel Co.* Production tests have shown that Super La-Led machines 80 pct faster than standard screw stocks.

11—CUTTERS: Two shear type tools for cutting steel cable and wire rope have been developed by *H. K. Porter, Inc.* The No. 1 TN cutter cuts all cable through ⅜ in. diam and the large No. 3 TN cuts all cable through ⅝ in. diam.

12—AL FLUORIDE: This newest addition to the *Fisher Scientific Co.* reagent line contains a maximum of 0.005 pct chloride, 0.001 pct sulphate, and 0.008 pct iron.

13—MANDREL NIBS: Standard line of cemented carbide underground braze type mandrel nibs for drawing steel, aluminum and other materials is announced by the Carboloy dept., *General Electric Co.* The line includes 17 diameters and special nibs can be furnished to specification.



14—CONVEYOR FURNACE: Specially built conveyor furnace heats aluminum and brass billets prior to forging. It is possible to use the furnace for tempering, annealing, stress relieving, or other heating operations requiring a maximum temperature of 1650°F. *Hevi Duty Electric Co.*

15—TRAY-CAST MACHINE: This machine is a production casting, molding or forming unit for materials such as melted ferrous or nonferrous metals, plastics, etc. Advantages of the arrangement this machine uses are the flexibility of pouring different types of molds at the same time by being able to charge molds into the machine within a short period of time. *Ferguson Equipment Corp.*

16—NI-AG STRIP: Eighteen percent Ni-Si strip, rolled to close tolerances and thin gages and foils, is available from *American Silver Co., Inc.* It is custom rolled in strip up to 6 in. wide and down to 0.0005 in. to tolerances as close as ±0.0001 in.

17—CLEANING COMPOUND: A noncorrosive, ferrous metal washing compound, for quick removal of quenching oils after heat treating is announced by *Ipsen Industries, Inc.* It is odorless and nontoxic.

Free Literature

20—FOAMGLAS: Publication lists condensed specifications for application of Foamglas in walls, floors, roofs, and in low temperature space insulation. *Pittsburgh Corning Corp.*

21—EMULSION CLEANERS: Specific industrial uses, maximum and minimum dilution ratios, and methods of application are several points covered in pamphlet A106 issued by *Turco Products, Inc.*

22—THERMOMETER BULBS: Catalog 5701 describes resistance thermometer bulbs of high speed, marine, room temperature and sanitary types, for temperature spans as narrow as 20°F. *Minneapolis-Honeywell Regulator Co.*

23—BUSHINGS: Price list and technical data on drill jig bushings are contained in bulletin 1102 issued by *Ace Drill Bushing Co., Inc.*

24—ZIRCONIA: Folder describing stabilized zirconia is available from *Zirconium Corp. of America.* Development of the process is explained as well as the oxide's unusual characteristics. Complete physical and chemical properties are covered.

25—ELECTRIC FURNACES: Bulletin covering electric heat treat furnaces has been published by *Holcroft & Co.* Four types of electric heating elements and methods of mounting are described.

26—MAGNETISM: Magnetism in cast stainless steel is the subject of data sheet stainless 3 obtainable from *Empire Steel Castings, Inc.*

27—GRINDING WHEEL: New principle for light grinding and metal finishing has been introduced by *Merit Products, Inc.* Grind-O-Flex is described in pamphlet.

28—CONTROL INSTRUMENTS: Catalog describing process control instruments is announced by *Fischer & Porter Co.* A variety of flow meters, recorders, controllers, pressure regulators, sight flow indicators, and chemical feeders are listed.

29—NI ALLOYS: Physical and mechanical properties of Inco nickel alloys are summarized in handbook available from *Alloy Metal Wire Co., Inc.* The 37-page book is a guide to wire, rod, and strip.

30—GRAY IRON: Bulletin A-174 is a reprint of article *Development and Control of Engineering Gray Cast Irons.* By applying proper principles, as cast acicular iron may be produced having tensile strengths of 60,000 to 80,000 psi; when heat treated, up to 105,000 psi. *International Nickel Co., Inc.*

31—STEEL CHART: Slide chart giving facts on steel casting material selection has been developed by *Lebanon Steel Foundry.* Reference data on 19 carbon and low alloy grades and 17 stainless and corrosion resistant grades in regular production at the company are given.

32—HEAT PUMP: All electric heat pump is described in bulletin available from *Westinghouse Electric Corp.* This is a complete self contained system that provides warm filtered air and cool, dehumidified air, without water or flame.

33—CASTING IMPREGNATION: Booklet on the subject of casting impregnation has been issued by *Polyplastex International, Inc.* Case histories of successful applications of impregnated castings are given.

34—FORGING: Smooth hammered forgings, composite die sections, and cast to shape tool steels produced by *Allegheny Ludlum Steel Corp.*, are described in bulletin printed by firm. Shapes available, weight limits, and analyses are outlined. A guide on uses and types available is also included.

35—SE RECTIFIER: Heavy duty selenium rectifier specially designed for use in plating and other metal finishing operations is described in bulletin offered by *Hanson-Van Winkle-Munning Co.* Information on models available, costs, operating capacity, type of input and kind of current switching is included.

36—CHARGING MACHINES: Brochure covering auto floor furnace charging machines, designed in eight models for handling raw materials and shapes that weigh up to 20,000 lb total maximum has been issued by *Salem-Brosius, Inc.*

37—MELTING FURNACE: Bulletin has been issued by *Lindberg Engineering Co.* on its line of Simplex melting furnaces. Photographs, specifications, diagrams, performance data, and other technical data are given.

38—SI RUBBER: Literature describing injection molding of silicone rubber products has been issued by *Minnesota Rubber & Gasket Co.* It defines the properties and applications of silicone rubber.

39—INDUCTION FURNACES: Large capacity, low frequency induction melting furnaces is the subject of brochure available from *International Selling Corp.* Standard type furnaces and essential operating data are shown and general layout of a low frequency zinc melting furnace plant and an aluminum furnace plant.

Journal of Metals
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March

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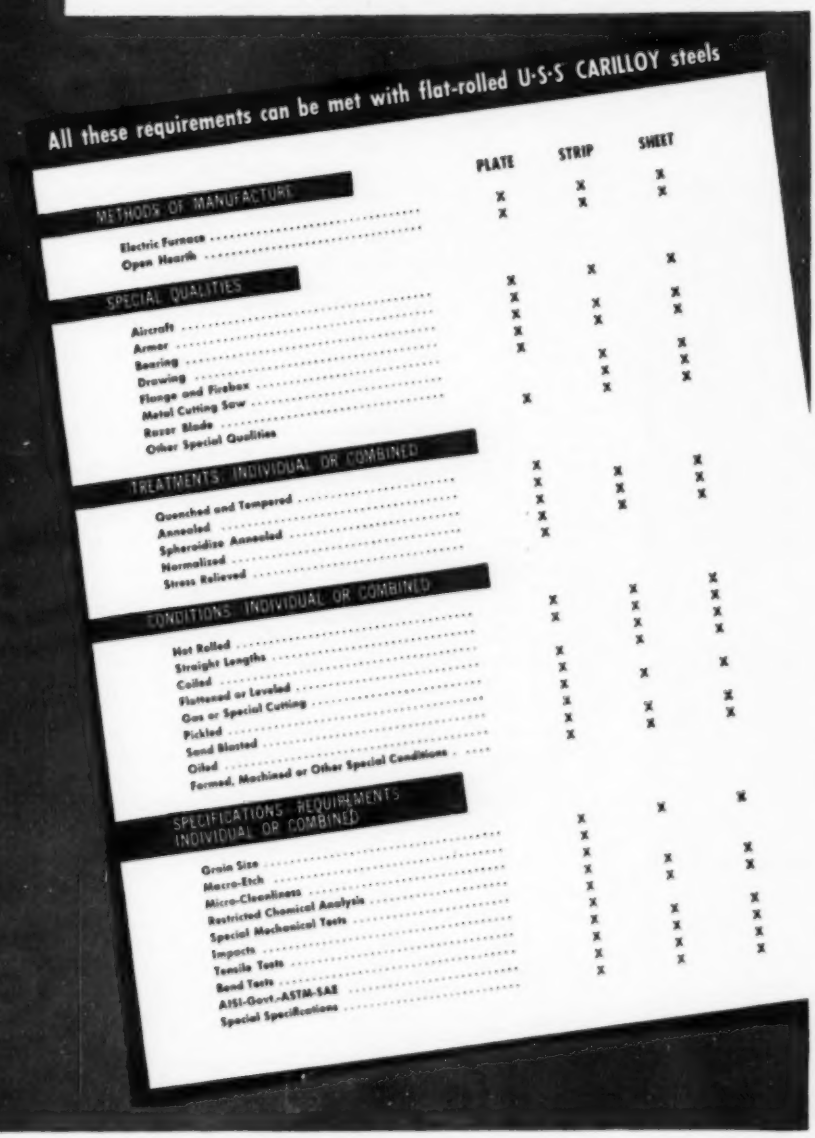

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JUST 10 min before launching time the sun cut through the fog, making the gray and black hull appear less ominous. Mrs. Dwight D. Eisenhower smashed the usual bottle of champagne over the port bow, and the USS *Nautilus* slid down the ways into the Thames River. A half hour after the world's first atomic powered submarine had been launched it was tied up at General Dynamics Corp. wet dock and workers once again swarmed over it, getting on with the job of finishing her.

The world's first nuclear powered submarine, the *Nautilus*, slides down the ways at General Dynamics Corp., Groton, Conn. shipyard.

When her atomic power plant is in place the *Nautilus* will be able to travel 20 knots per hr underwater—and stay under as long as her personnel can stand the strain of confined quarters. Unlike other subs, the *Nautilus*' engines operate independently of oxygen. The atomic sub will also be able to travel around the world without refueling.

The *Nautilus* engine, Mark II, will be installed in time for the submarine to make her first runs sometime this summer. The Prototype Mark I, is currently operating on dry ground at the National Reactor Testing Station in Idaho. Westinghouse Electric Corp. is responsible for construction of both engines. Assembly of the Mark II will take place inside of the hull of the *Nautilus*.

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DESPITE the close relationship between American youth and the pseudo science of space travel, ray guns, and hot rods, results of a recent survey released by the National Education Assn., indicates that youngsters are passing up school science courses. They think them dull and unrealistic.

According to the survey taken in schools with a total enrollment of 326,000 students in 42 states, science teachers feel that the shortage of engineers could be alleviated if the importance of modern technology and the opportunities in that field were driven home more strongly to the students.

Why students don't choose science courses was a question posed by the survey. Some of the answers were: Reluctance of students to take courses which are somewhat more difficult and which might lower their grades; lack of communication between high schools and colleges concerning science programs; little emphasis on science experience by teachers to students in grade schools.

Following somewhat the same line, *Mineral Industries*, published by Pennsylvania State University, did a figurative disection of metallurgy students—seeking individual reasons for choice of career. Hal Lee Harman, third semester metallurgy student gave four reasons for going into metallurgy. They were: So long as our present civilization persists, metals will have to be won from their ores, refined, and shaped; the current expansion of the metals industry is creating new opportunities; he was interested in metals; and he thought he had capabilities suited to the profession.

Charles McHugh, another student, was uncertain as to career when he finished high school. School experience had highlighted strong interests in chemistry and engineering. He lived in a Pennsylvania coal mining community, near steel producing Johnstown. Thus, he narrowed his choice to fuels or metallurgy. College experience brought the final choice down to metallurgy. J. Richard Kirn lives in Philadelphia where his father operates a small nonferrous foundry. The foundry had much to do with his final decision.

Mineral Industries states that "in the high schools of the state (Pennsylvania) students have little chance of studying in, or even hearing of, such exciting fields as geology, geochemistry, metallurgy, fuel technology, mineral economics, or mineral preparation."

Finally, from Harvard University, 23 prominent U. S. educators warn that the nation is critically near a breakdown in the first step of training scientists—high school science teaching. They find that the situation is "not only insupportable but perilous."

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INTERLAKE Iron Corp. engineers H. W. Campbell and C. P. Johnson, respectively chief engineer and Chicago district superintendent, recently presented a paper at the Eastern States Blast Furnace & Coke Oven Assn., in which they told of revolving the 35-ton hopper at the top of a blast furnace at better than 20 rpm. They report more even distribution of iron ore, coke and limestone in the furnace.

Revolving tops made their first appearance in the iron and steel industry during the early 1900's with the 6-point revolving distributor. However, as furnaces began using double-skips for charging, receiving hoppers were designed to accommodate off-center skip dumping. Low and high spots were created in the pile in the bell by swirling motions said to be motivated by the design.

Campbell and Johnson conceived the idea of having the small bell hopper revolve at a fast clip during skip dumping. With the top spin fast enough to perform at least one revolution in the time need-

ed to clear a skip, piling should be level on the small bell, and level when dropped on the big bell. It also followed that by alternating the direction of rotation so as to oppose the feed swirl direction it would be possible to increase the relative speed of hopper rotation. Same general mechanical design for the system is used as for the conventional top. Motor size is 35 hp and a lower ratio speed reducer is needed. Control, because it eliminates the 6-point feature, is simple. As the skip goes into slowdown at the top of the furnace, the hopper starts to spin. By the time the skip reaches dumping position, the hopper has reached full speed. Rotation reversal for each skip is simple.

Patents were applied for, and in ordering a top for the new Erie stack, the 6-point top was completely ignored. Interlake is making the high speed tops available to the industry through licensing arrangements.

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GAW stands for guaranteed annual wage—and there are indications that GAW will be one of the main objectives of the CIO's United Steelworkers when the union begins contract negotiations in a few months. A recent issue of *Steel Labor*, the union's official publication, maintained that GAW is a "basic, practical way" to meet unemployment and to head off a "more serious downturn in the years to come."

The article continued that "just as the steelworkers pioneered the pension and social insurance programs that set the pattern for industry throughout the country, so we propose to lead the way in the fight against unemployment and depression with the guaranteed annual wage."

United Steelworkers made a demand for a guaranteed wage in 1952, but compromised by accepting a wage increase. A GAW proposal submitted to Aluminum Co. of America last year called for a weekly amount 30 times the standard hourly rate in the event of a worker's unemployment. It was turned down. The Steelworkers and the International Electric Workers, another CIO union, have joined in sending out questionnaires dealing with layoffs over the past 16 years to leading corporations. Among those receiving the questionnaires are U. S. Steel Corp., Bethlehem Steel Corp., and General Electric Corp.

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ALUMINUM Co. of America's concern over long term power arrangements for its Vancouver and Wenatchee plants in the Pacific Northwest may result in the company spending its own money to build hydroelectric power dams.

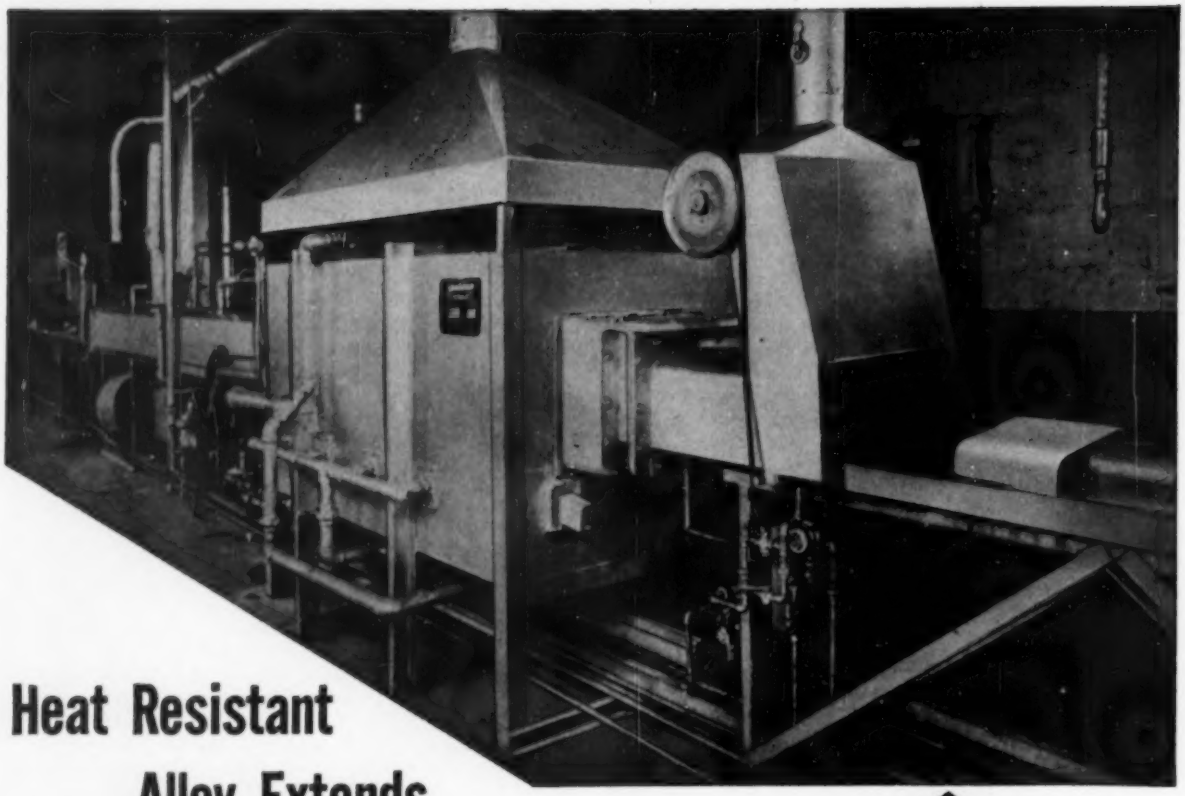
C. S. Thayer, northwest manager of Alcoa, says that "informal conversations in many quarters" have already taken place in reference to getting more generating power with Alcoa's help. A significant amount of hydroelectric power for aluminum plants is currently on an interruptible basis, delivered when available, but during short periods of supply, when rivers are low, likely to be cut off.

Mr. Thayer said Alcoa would work with private utility companies or with public utility districts, as demanded by the situation. Thus far the talks have been extremely tentative and solely with private utilities.

Mr. Thayer states that Alcoa has stayed out of the political controversies surrounding power development, the main objective being to assure firm power "at a cost competitive with other locations more favorably situated as to transportation and other factors."

With $13 million worth of facilities built or authorized for the fabrication of aluminum products in the Northwest, Alcoa "will be ready to invest additional money in new plant fabricating facilities as soon as the company can assure itself that its present aluminum plants have adequate long-term supplies" of economic electric power. The company "is ready and willing to consider investing its own money in any suitable manner that will assist in expanding the supply of power in the Northwest and at the same time maintain a sufficiently economical power cost for its production of aluminum."

A heat pours from the 20 ft diam, top charged electric furnace at the Sheffield Steel Corp. plant at Houston, Texas. Sheffield is a subsidiary of Armco Steel Corp. The furnace has been operating for a little more than one year.



Heat Resistant Alloy Extends Equipment Life

Still good after 14 years

Original Muffle ...cast in a 35% nickel-18% chromium heat resistant alloy, trade-named "Chromax," and produced at the Harrison, N. J., plant of Driver-Harris Company ... still serves after 14 years in this gas fired, controlled atmosphere, pusher-type furnace manufactured by Sunbeam Corporation, Industrial Furnace Division, Chicago 23, Ill.

The muffle of this annealing furnace has withstood continuous alternate heating and cooling since 1940, except for a shut-down period during the Korean war.

Still in use 14 years after installation, this casting... produced in 35% nickel-18% chromium heat resistant alloy... demonstrates the economy of using nickel alloyed material to resist growth, warping, scaling and other deteriorating effects of heat.

From among the many engineering alloys containing nickel, you can select a material to provide the best set of properties for meeting one or a combination of requirements.

Let us help you find practical solutions to your metal problems. Make use of our counsel and data based on years of specialized experience in the fabrication, treatment, properties and performance of alloys containing nickel. Send us details of your problems for our suggestions.

Heat Resistant nickel-chromium alloy is also used for the annealing furnace conveyor.

INCO

THE INTERNATIONAL NICKEL COMPANY, INC. 67 WALL STREET NEW YORK 5, N.Y.

Chemical Construction Corp. signed an agreement with Calera Mining Co., a subsidiary of Howe Sound Co., to manage and operate Calera's new cobalt refining plant at Garfield, Utah, for a period of two years. Chemical Construction is a subsidiary of American Cyanamid Co. The plant has been in partial production since late 1952. Object of the contract is to enable Chemico, designer and builder of the plant, to place the operation on a commercial basis. Calera will supply cobalt concentrate from its Blackbird mine and mill in Idaho for processing by Chemico on a toll basis. The plant is the first to embody Chemico's chemical technique for recovery of cobalt metal from ore concentrates.

A certificate of necessity for $55.25 million was issued to the Nicaro Nickel Co., subsidiary of Freeport Sulphur Co., for facilities at Moa Bay, Cuba, and New Orleans. The certificate will allow the company to write off 80 pct of the estimated cost in five years in depreciation for Federal tax purposes. Freeport plans to spend $35 million on Moa Bay mining operations and $18 million on a pilot processing plant near New Orleans. The remainder of the funds will be used for ore carriers. Office of Defense Mobilization also announced allocation of $43 million to National Lead Co. for expansion by 75 pct of the government-owned nickel plant at Nicaro, Cuba.

Alumina Jamaica Ltd., Aluminium, Ltd., subsidiary, sent its first bulk shipload of alumina to Norway. Previous shipments had been in 100 lb paper bags, but with output expanding as construction of the $30 million plant, transport, and port project nears completion, the company has changed to bulk shipment. By summer it is expected that a large share of production will go through the Panama Canal to Kitimat, B. C., on the west coast of Canada.

Iron and titanium ore in what was termed substantial amounts but of unknown commercial value has been found on land partially owned by the Union Pacific Railroad. The road discovered 178 million tons of the ore near Laramie, Wyo., in 1952, according to William Reinhardt, Union Pacific vice-president, and drilling increased reserves to 228 million tons. Much of the material is low grade he said, but part of it was found to average 46 pct iron and 19 pct titanium.

Government has again postposed its decision on a third round of aluminum expansion. Defense Production officials had recommended a 200 million lb expansion in domestic primary aluminum capacity more than a year ago. Most recent statement from Office of Defense Mobilization is that a check made by the Defense Dept. of the aircraft industry shows "a sufficient number of undetermined factors to necessitate a review of the entire military requirements for aluminum."

Drift of Things

IT is a rare occasion when we are privileged to see History being made, and when we left for Groton, Conn., early one morning a few weeks ago, the heavy fog and mist failed to dampen our spirits. We boarded the "Nautilus Special," an extra train added to bring New Yorkers direct to Groton, and became part of the 12,000 people that would see the launching of the world's first atomic submarine. Only person we recognized on the way was Oliver Ralston of the U. S. Bureau of Mines and we were able to spend a few minutes talking to him. (On the return trip we had a long talk with Henry Hausner of Sylvania Electric Products, and Mrs. Hausner).

Not in our train, but traveling in a special one of her own, was Mrs. Dwight Eisenhower, with a party of Naval officers and government officials. We all arrived at about the same time, and the launching ceremonies began.

As at all launchings, there were speeches. John Jay Hopkins, chairman of the board and president of General Dynamics Corp., the company that built the submarine; Gwilyn A. Price, president of Westinghouse Electric Corp., the company that built the atomic power plant; Admiral Robert B. Carney, chief of Naval Operations, the organization that will run the sub; and Lewis L. Strauss, chairman of the U. S. Atomic Energy Commission, the organization that will watch over all the others, spoke seriously and briefly. Rear Admiral Edward B. Harp, chief of chaplains, USN pronounced the benediction.

Between the benediction and the time Mrs. Eisenhower stepped to the launching platform, as if as an omen of a bright future, the heavy fog began to dissolve, and the sun took over, illuminating the scene. At precisely 10:57 am, Jan. 21, 1954, Mrs. Eisenhower named the submarine *Nautilus*, and turned it over to Commander Wilkinson, its prospective skipper. The great submarine outshone all speakers and visiting dignitaries as she slid down the ways into the water.

Since most of the details of the new submarine were given wide coverage in the newspapers, only some of the lesser known ones will be covered here. The *Nautilus* engine, the Mark II, will be the first ever to provide the motive power for any vehicle or vessel by controlled nuclear fission. This power plant could furnish enough electricity to meet the needs of a small city. The *Nautilus* will be capable of crossing the Atlantic Ocean submerged and at full speed. It is said that if it were not for the endurance limits of the men that will man her, the *Nautilus* could circumnavigate the globe without surfacing.

The keel for the *Nautilus* was laid on June 14, 1952 with the then President Harry S. Truman officiating. His initials are bead welded onto the ship's keel plate. If we remember correctly, a keel plate is submerged even when a submarine is surfaced.

The *Nautilus* is 340 ft long, weighs 3000 tons, and cost $55,000,000. That would be $18,333.33 per ton ($9.16 per lb) or $161,764.71 per ft, expensive any way you look at it. Appropriately enough, domestic champagne was used for the christening.

WE certainly will never oppose progress, but we just can't face up to the possibility of living in the age when "the car of the future" becomes a reality. There is so much that can be done to make today's automobile more practical, while at the same time increasing safety and beauty. The designers are obsessed with the notion of speed and individuality. The end result, however, seems to be that all futuristic cars look like copies of each other.

It is recognized that there is a growing cult of race car and sport car fans. In their proper place, with racing done in selected areas, this group can accomplish much in the way of automotive improvement, and at the same time give expression to a lively out-of-doors hobby. If the trend were for everyone to participate, we feel that the insurance rates would result from everyone driving the new 19?? cars would bring driving back to the days when it was for a privileged few.

Getting back to today's automobiles, we must admit that the designers are considering safety factors. At a recent exhibition we saw a block long limousine which featured a built in bar in the backseat arm rest. The designers had wisely concluded that it would not be safe to locate the bar too near the driver.

A. S. Cohan

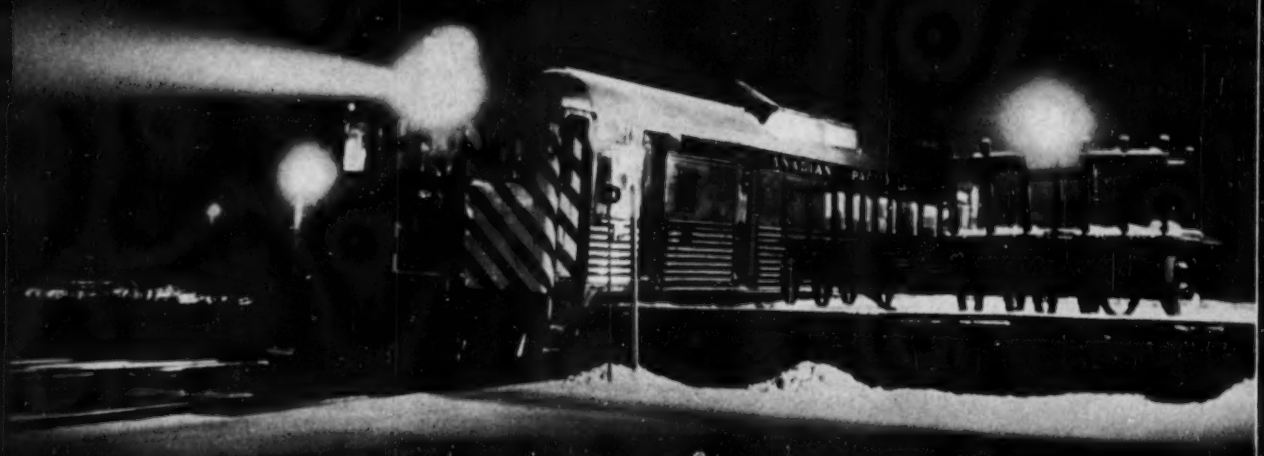
EACH year the Secretary has the pleasant duty of informing two or three dozen AIME Members that they have completed 50 years of continuous membership and thus are privileged to be guests of the Institute at the Annual Banquet to receive a certificate and pin as members of the Legion of Honor. Letters in reply are often deeply appreciative of what the society has meant to these elder statesmen. George W. Bryant has just written, "I recollect with pleasure my long membership in the Institute and I consider that it has been of great value both professionally and socially, and I hope that young engineers in the future will look upon it in the same way." And from a 50-year Member in South Africa: "My membership in the Institute has been an inspiration and a source of pride and pleasure."

Most of these Members who hang on for 50 years have been active in Institute work. They are good examples of a contention of ours that what one gets out of the Institute is in geometrical proportion to what one contributes, not as money, but as talent, interest, and effort.

Occasionally our efforts to reach a prospective Legion of Honor member unearth some disquieting news. Back in 1945 for instance, we tried to reach a man in England who had joined in 1895. Not being successful, we tried the indirect approach and asked another of our English members if he could tell us where the gentleman in question was. He replied as follows: " . . . has been dead for a good many years. . . . went to the bad, rather, after reaching England, and I can only suggest that you write him on asbestos paper, and address your letter in care of the Devil." (E. H. R.)

LIGHT IN THE NIGHT...

Spearing across the snows
of Canada shines this headlight.
But for refractory brick,
the arc wouldn't spark to flash
the headlight, the stainless
steel with which the car is built
would not exist . . . the trucks
she rides on, the motors and the
generators, even the glass . . .
depend for their existence upon
quality refractories
such as Grefco supplies
the world.



Budd Rail Diesel Car on the
Canadian Pacific Railway

GENERAL
REFRACTORIES
COMPANY
Philadelphia

Products of flame. Wherever there's flame

GENERAL REFRACTORIES COMPANY

Copper in Sweden—

Copper Smelting in Boliden's Rönnskär Works Described

As much of the production as possible in Ronnskar works is handled during the day shift. This requires three times as large furnaces and casting machines as three shifts and involves a considerable outlay of capital, but the labor force is reduced approximately two thirds with production costs decreased to a corresponding degree.

by Olov Herneryd, Olof A. Sundstrom, and Allan Norro

IN 1942 a body of ore containing considerable amounts of gold, silver, copper, and arsenic was discovered at Boliden, a small community in northern Sweden in the province of Västerbotten, and mining was started in 1926. At that time, however, no copper works existed in Sweden capable of working this type of ore, and quantities of ore containing a relatively high degree of gold were sent to foreign smelters, among them the one at Tacoma, Wash. Later, additional deposits containing not only copper but also lead and zinc were found and mined. At the present time the total annual ore production is about 1.2 million metric tons.

The ore is generally treated at flotation mills built close to the mines, and the resulting concentrates are transported by truck or cable railway sometimes to Boliden and from Boliden by rail to Rönnskär, and sometimes to other railway stations whence it is forwarded to Rönnskär. Here the copper and lead concentrates are worked together with concentrates and scrap from other parts of Sweden and abroad, while the zinc and pyrite concentrates are shipped to customers in and out of Sweden. Table I gives the production of the Boliden Mining Co. in 1952.

The construction of the Boliden smelter, the Rönnskär works, was started in 1928 and production began in 1930. The plant is situated on a peninsula in the Gulf of Bothnia approximately 55 kilometers from the Boliden mine. This site was selected partly because it offered good opportunities for a harbor and partly because through locating the works as far out in the sea as possible the damage on surrounding forests and agricultural districts by the waste gases could be minimized.

The original plant was built with special consideration of the type and quantity of the ore in the Boliden mine. The ore is fairly poor in copper but rich in gold and arsenic for which the flotation methods of that time could not be used. When production was started in 1930 the equipment of the smelter consisted of four small Lurgi roasters, one silica lined reverberatory furnace with interior dimensions 5x24 m and two small Pierce-Smith converters, 2.0x2.2 m.

The smelting capacity which at first was rather small was rapidly increased, as shown in Table II. At the same time new departments such as electrolysis and precious metal works, and a cathode furnace, and also new furnace units were added.

Since there are no coal deposits of any consequence in Sweden, the operation of the reverber-

O. HERNERYD, O. A. SUNDSTROM, and A. NORRO are respectively General Manager of Ronnskar Works, General Superintendent of Copper Works, and Superintendent of Copper Smelter of the Boliden Mining Co., Skelleftehamn, Sweden.

Table I. Production of Boliden Mining Co. in 1952

Material*	Metric Tons
Iron pyrites	360,000
Zinc concentrates	15,000
Electrolytic copper	29,000
Refined lead	12,000
Arsenic products	15,000
Gold	3.66
Silver	18.50

* Selenium, bismuth, and lead compounds are also produced.

atory furnace depended entirely on coal imports. Under the pressure of international political events in 1938 and 1939 and the anticipated danger of being cut off from coal imports, a change over to electric smelting was being discussed before the outbreak of war. However, it was not until 1946 that it was decided to design and build an electric furnace and to modernize other departments of the copper works.

The Rönnskär works was modernized with the following principles in mind: A—Maximum consideration given to the hygienic problems created by the presence of SO_2 and As_2O_3 formed in the different processes, B—the processes should be conducted in as large units as possible, C—mechanization should be developed as highly as possible, and D—as much as possible of the production should be handled during the day shift, e.g. all casting. These principles require three times as large furnaces and casting machines as production with three shifts and involves a considerable outlay of capital,

Table II. Smelting Capacity and Analysis of Matte and Slag

Year	Solid Charge Metric Tons	Analyses of Matte			Analysis of Slag					
		Au g per ton	Ag	Cu pct	Au g per ton	Ag	Cu pct	SiO_2 pct	FeO pct	CaO pct
1930	26,500	160	400	27.0	0.5	10.0	0.33	35.9	40.4	6.0
1935	268,000	96	322	11.2	0.9	6.0	0.25	34.7	48.7	1.7
1940	221,600	128	465	18.3	1.0	7.0	0.33	32.7	48.8	2.9
1945	192,200	46	418	38.4	0.3	4.0	0.49	33.7	46.5	2.1
1950	178,600	28	307	36.0	0.3	2.0	0.40	35.8	38.1	4.3
1951	168,700	28	281	36.2	0.2	2.0	0.34	38.9	38.2	4.1
1952	174,500	29	309	34.6	0.3	3.0	0.31	38.8	41.5	4.0

but the labor force is reduced approximately two thirds with production costs decreased to a corresponding degree. The result is that the Rönnskär works has in many cases as large production and transportation units as American plants although the production is no more than 1/10 of the American works.

The modernization of the smelting furnace and converters resulted in a certain overcapacity. This overcapacity has been utilized to increase the production mainly by treating foreign material on a custom basis.

Roaster Dept.

The material from the Boliden mines consists of three radically different types of concentrates: A—Copper concentrates with approximately 25 to 27 pct Cu and more than 30 pct S and 0.5 to 2.0 pct As, B—copper concentrates with 25 to 27 pct Cu with 30 pct S and 2 to 6 pct As, and C—concentrates with less than 1 pct Cu but relatively rich in gold and with 20 to 25 pct S and more than 10 pct As.

On arrival, the smelting material is separated with regard to the arsenic content into bedding bins. The copper works has four such bins each holding about 4500 metric tons, with conveyers for the transportation of the material to and from the bins. The raw material and flux are stored in relatively thin horizontal layers in a proportion suitable for smelting. As the pockets are emptied through openings at the bottom more or less vertical layers are achieved thus giving a fairly satisfactory mix-

Fig. 1—Current is conducted to each electrode via the electrode clamps. These clamps are water cooled and are kept in position by screws through a cast iron ring.

ture of the material before it is taken by conveyers to pockets above the roasters.

Because of the high arsenic content in some of the material special precautions have been taken to bring down the amount of gas and dust in the roaster building to a safe degree from the point of view of hygiene. For this purpose each roaster is supplied with a ventilated hood on top and in addition a drum on the furnace wall opposite each row of doors gathers gas and dust which otherwise could penetrate when rabbles and arms are changed.

The calcine is gathered in containers holding about 4.5 metric tons. The connection between roaster and container is so designed that dust and gas cannot escape into the building. The small amounts of calcine which gather at the top of the containers are washed off and collected.

The roaster gases are conducted via cyclones in which considerable amounts of metal-containing dust are separated and immediately returned to the furnace by a screw conveyer. The roaster gases then go to a gas purifying plant of conventional con-

Fig. 2—Electrodes used in the electric smelting furnace are of the Soderberg type. Approximately one metric ton of Soderberg paste is emptied into the electrode.

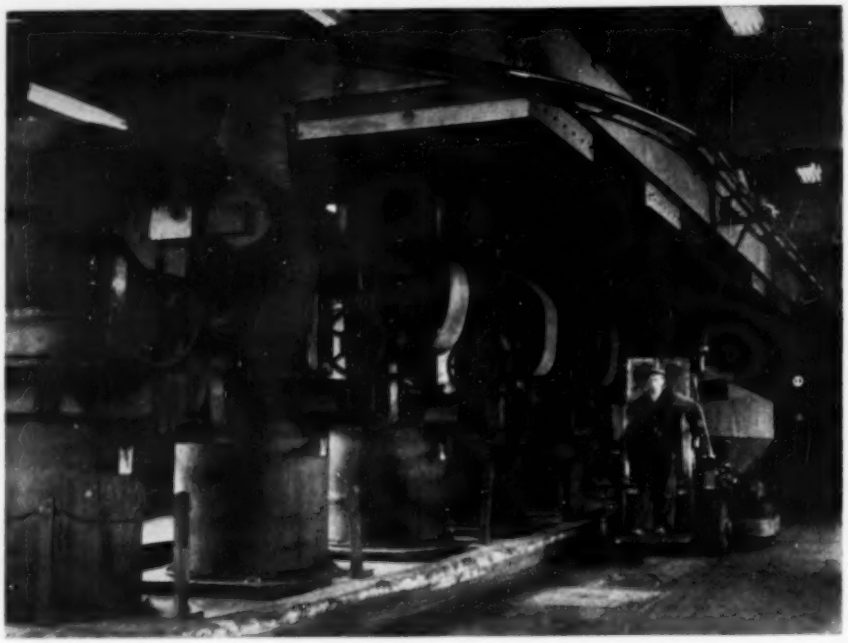


Fig. 3—When the electrode is burnt short and normal travel is no longer possible, the electrode is lowered by means of the Wisdom-bands welded to the sides of the electrode. The furnace is charged by emptying containers from the roasters into charging pipes.

struction consisting of brick-lined dust chambers and air coolers made of iron pipes and Cottrells.

The material containing the highest degree of arsenic, the arsenic concentrates, is roasted in special furnaces and together with other compounds and the resulting As_2O_3 is precipitated in the gas cleaning plant and collected as crude arsenic containing up to 95 pct As_2O_3. This is transported to concrete silos for storage. The average As_2O_3 content is, however, lower, as shown in Table III.

The copper concentrates with an arsenic content of between 2 to 6 pct are also roasted separately. The arsenic-rich dust containing comparatively much metal, is conveyed to the roaster where the arsenic concentrates are roasted. In this manner it has been possible to bring down the metal losses in the resulting crude arsenic. The majority of the copper concentrates with an arsenic content less than 2 pct is merely dried in one of the roasters. From 150 to 200 metric tons are roasted in each furnace daily and Table IV shows analyses of input and output material.

Electric Smelting Furnace

The electric smelting furnace with an inside area of 6x23.5 m has six Söderberg electrodes in a row each with a diameter of 1.2 m, and six transformers each of 2000 kva. The six transformers are divided into three groups, each group having two transformers in parallel and connected with two electrodes. Consequently the furnace can be said to consist of three one-phase furnaces while the electrodes are Delta connected.

Table III. Average Analysis of Crude Arsenic Precipitated in Gas Cleaning Plant

Grams Per Ton		Pct						
Au	Ag	Cu	Ni	Pb	Bi	Sb	Se	As_2O_3
3.0	18.0	0.4	0.01	0.1	0.07	0.4	0.18	81.1

The electric connection between the transformers and the electrodes consists of eight 325x10mm interleafed copper busbars per electrode. From their ends the current is conducted via 48 flexible cables per electrode and firm, thick, water-cooled copper tubes to the electrode clamps as illustrated in Fig. 1. These are water-cooled and kept in position and pressed against the electrode by screws through a rigid watercooled cast iron ring. Because of the special manner of the interleafing of busbars, it has been possible partially to conduct them between the respective pair of electrodes so that the distribution of current via the flexible cables can be made from both sides of the electrodes. Consequently the length of the cables, which of course cannot be interleafed, is comparatively short. That this construction is good has been shown by the fact that the power factor, measured not by cos ϕ — m but

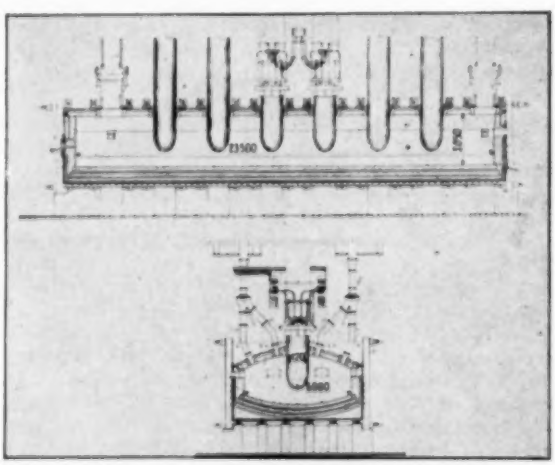


Fig. 4—The electric copper smelting furnace has an inside area of 6x23.5 m, six Soderberg electrodes each with a diameter of 1.2 m, a capacity between 500 to 700 metric tons, and six transformers each of 2000 kva.

through reactive effect measuring, even when the furnace is utilized to highest capacity reaches very high values as shown in Table V.

The furnace transformers are primarily fed with 30,000 v. On the secondary side, a range of 17 tappings is provided within each of the following voltage intervals to suit different furnace conditions: 50 to 100 v, 100 to 200 v, and 200 to 400 v.

In addition an interval of 150 to 300 v is provided for one group of transformers. The 17 tappings are brought out to a selector switch of the on-load type, motor operated, and arranged for remote control by the furnace operator. These voltage intervals are obtained by changing the connection of the busbars to the transformer secondary terminals.

The electrode clamps were originally cooled with cool water. However, it often appeared, particularly in winter time, that hollows were created in the electrodes between the clamps and the coked part of the electrode below the arch of the furnace.

Table IV. Analysis of Concentrates Before and After Roasting in 1952

Material	Pct As	Pct S
Copper Concentrates		
Concentrates	1.80	34.2
Calcine	0.36	14.0
Arsenic Concentrates		
Concentrates	11.0	21.0
Calcine	1.37	13.9

These hollows were often so large that they covered up to ¾ of the electrode area with great danger of electrode breakage. Now, by permitting the cooling water for the clamps to circulate, the temperature of the water has risen to between 50° and 60°C. After this change was made no more hollows were found. Also by using warm water for the electrode very little tightening of the pressure screws for the clamps is required to obtain perfect contact with the electrodes, and the shape of the electrode below the clamps becomes practically cylindrical which greatly facilitates the sealing between electrode and gland in the furnace roof. Because of the better contact between clamps and electrodes the electric loss here has been brought down to only 0.3 pct of the total power input when operating at full capacity as compared to 1.5 pct or more previously.

The electric power input to the furnace is regulated from a control room on a level just above the furnace arch. It is possible to regulate the power input both automatically and manually, but in practice the voltage is always manually regulated. The automatic effect regulation is based on a constant resistance between electrode and matte. A balance relay, effected by the current and voltage of an electrode, is the controlling device in combination with a rheostat in the voltage circuit for achieving the desired position of the electrode. The effect regulation is obtained in principle through adjusting the position of the electrode in comparison with the level of matte. Therefore, each electrode has a winch by which it may be elevated or lowered.

In comparison with other common systems this method of regulation has certain advantages. It is not only possible to vary the power input, i.e., the heat generation, in the longitudinal direction of the furnace, but also to concentrate it to different levels

Table V. Measurement of Power Factor for Electric Smelting Furnace

Voltage Between Electrodes No. 1 and 2	3 and 4	5 and 6	Active Power MW	Reactive Effect MVAr	Cos φ
276	255	280	10.8	2.4	0.976
280	265	285	10.0	2.1	0.979
290	260	290	8.0	1.4	0.985
290	270	293	6.0	1.0	0.986
290	270	300	4.0	0.6	0.989
230	230	240	4.0	0.6	0.989
230	225	235	6.0	1.17	0.982
221	220	230	8.0	1.98	0.971
220	215	222	10.0	2.85	0.963
209	202	210	11.9	4.50	0.935
180	193	190	4.0	0.79	0.981
165	188	186	6.0	1.65	0.964
175	180	180	8.0	2.85	0.942
168	173	168	10.0	4.70	0.905

in the molten slag. This makes it possible to maintain a protective layer of magnetite on the bottom of the furnace below the electrodes. If this layer becomes too thick, the electrodes are lowered increasing the bottom heat, and if the layer becomes thin, the electrodes are raised generating heat closer to the surface in the furnace. The furnace operation is, in other words, highly flexible.

The electrodes are of the Söderberg type as shown in Fig. 2. The Söderberg paste is smelted in a pot which is heated by circulating hot air, thus avoiding any danger of over-heating. This pot can then be transported by crane to a point above each electrode and the charge, approximately one metric ton at the time, is emptied directly into the electrode. There is no subsequent ramming of the electrode. When the part of the electrode dipping into the slag has become burnt so short that normal travel of the electrode is no longer possible, the clamps are loosened so that the electrode is suspended only from

Table VI. Electric Smelting Furnace Production Data for 1950, 1951, and 1952.

Data		1950 Quantity*	1951 (11 1/3 months)	1952 (9 months)
Total charge, exclusive of fluid converter slag	ton	178,555	159,940	133,720
Calcine, 700°C hot	ton	88,100	76,600	65,600
Dried concentrates, etc.	ton	73,410	70,830	55,525
Cold return material	ton	17,045	12,510	12,595
Copper in charge	pct	16.00	15.49	14.44
Matte produced	ton	84,200	72,870	61,200
Copper in matte	pct	36.0	36.4	34.4
Avg temp of matte at tapping	°C	1090	1085	1065
Amount of slag	ton	142,000	128,000	107,000
Copper in slag	pct	0.40	0.34	0.31
SiO_2 in slag	pct	38.8	38.9	38.7
FeO in slag	pct	38.1	38.2	41.7
CaO in slag	pct	4.3	4.1	4.5
Ratio $\frac{O_2 \text{ in } SiO_2}{O_2 \text{ in FeO}}$		2.44	2.44	2.22
Avg temp of slag at tapping	°C	1185	1185	1195
Electrode consumption	kg per ton charge	1.44	1.57	1.78
Electricity consumption	kwhr per ton charge	389	398	417
Average power input	kw	7956	8417	8906
Power input per sq m of hearth area	kw	56.8	60.1	64.3
Labor hours per ton cold charge from the transportation of the roasted material to the furnace up to and including the tapping of slag and matte	hr	0.414	0.365	0.380
Labor hours for repair and maintenance	hr	0.10	0.11	0.11

* All tons are metric tons.

Fig. 5—Three tap holes for slag are located 1.0, 1.12, and 1.25 m above the lowest bottom level. Slag, which is tapped almost continuously, flows through covered and ventilated launders to a granulating plant.

two steel bands, 1.6x175 mm, the so called Wisdom-bands which are always welded to two opposite sides of the electrode. These pass through a brake shown in Fig. 3 and thus the electrode can be made to slip down slowly. As a rule an electrode must not be lowered more than 25 cm a day to be sure having the electrode safely coked.

The furnace itself is separated from the building and rests on a number of concrete columns. Around the columns a canal has been dug into the ground which connects with a larger hollow outside the furnace building. This hollow is so large that it can contain all the molten material in the furnace, about 500 to 700 metric tons. In case of a breakage there is no danger of damage to the building.

Sections of the electric furnace are shown in Fig. 4. The brick lining in the furnace closest to the bottom plates consists of fireclay of the Höganäs-type and is mainly intended as a filling material. Above the Höganäs-layer are three chamotte courses each 65 mm thick of a type called Bjuf F. Above the top layer is a sliding joint 20 mm thick of crushed magnesite and water glass; above them there are two magnesite courses (each course 250 mm thick) of Ankral-quality with a sliding joint 20 mm thick in between also of water glass and magnesite. The different courses were laid one by one and carefully dried before the next layer was laid. The drying was effected through covering the whole furnace bottom with a single coil of reinforcing iron forming a resistance to which a tension of 220 v was applied. On the walls closest to the cast iron plates there is a 125 mm thick course of chamotte, Bjuf F, and inside that two courses of magnesite, each course 250 mm thick. The magnesite brick covers the walls only up to a level in line with the normal surface of the slag and above this point the walls consist of chamotte-brick, Höganäs. On the outside the walls are covered with an isolating layer 125 mm thick to keep heat radiation losses from the furnace low.

In the arch are a large number of openings for the charging pipes, openings for six gas exhausts and, six openings for the electrodes, and finally one opening where the liquid converter slag is poured. The six gas exhausts join together in one pear-shaped flue whose bottom is covered with a wear-resistant layer of basalt-rock on which a chain conveyer glides. Thus, dust accumulated in the flue can be continuously returned to the furnace. The flue is connected with a Cottrell-filter where the last traces of dust conveyed by the gases are recovered. Volume of gas averages 30,000 cu m per hr.

The charging openings in the arch are placed with consideration to the drop angles so that the charged material will cover the whole surface with the exception of the part that is closest to the side where the slag is tapped, thus giving a settling zone here. The charging pipes are made of cast iron. At the point where they enter the furnace roof they are equipped with inspection openings.

The charging of the furnace is made through emptying the containers arriving from the roasters directly into the furnace via the charging pipes as shown in Fig. 3. The otherwise common pockets for roasted material are not necessary in this case since the furnace is so large that temporary fluctuations in the input of smelting material are absorbed and adjusted for by increasing or diminishing the amount of unmolten charge in the furnace.

On one of the narrow ends of the furnace are three tap holes for slag placed on somewhat different levels, namely 1.0, 1.12, and 1.25 m above the lowest bottom level. The slag, which is tapped almost continuously, flows through covered and well ventilated launders shown in Fig. 5 to the granulating plant where it is hit by a number of strong water jets. The mixture of slag and water flows to a container where additional water may be added. The resulting mixture of slag and water in a proportion of 1:10 is pumped from there by means of two Hydroseal-pumps connected in series through an 8-in. iron pipe of a maximum length of 600 m to the slag dumps; should the pipe line be longer one more pump can be and has to be connected in series with the two others. Each pump can produce approximately 8000 liters per min with a pressure of approximately 2 kg per cu cm. In this way it is possible to transport close to one metric ton of granulated slag per min. There is also an arrangement whereby the slag can be sampled continuously making for extraordinarily safe results which can hardly be achieved when the sampling is made by the spoon method. The granulating plant, which was constructed in collaboration with the Allen-Sherman-Hoff Pump Co., Philadelphia, was installed in 1950 and has functioned very satisfactorily. The wear on pumps and tubes is relatively insignificant.

On the opposite narrow end of the furnace are three tapping holes for matte placed as far down as possible. The one farthest down is only meant to be used in case the furnace has to be completely emptied. The two other tapping holes are used one at a time at intervals of one to two weeks. Normally the level of slag is approximately 120 to 130 cm over the lowest bottom level.

Table VII. Analysis of Electric Smelting Furnace Matte and Slag

Material	G Per Metric Ton		Pct							
Matte	30.5 Au	332 Ag	34.4	Cu	30.2	Fe	0.4	Ni	5.4	Zn
			24.6	S	2.7	Pb	0.3	As		
Slag	0.3 Au	3.7 Ag	0.31	Cu	32.4	Fe	0.03	Ni	4.1	Zn
			0.8	S	0.5	Pb	0.4	As	38.7	SiO_2
			8.1	Al_2O_3	4.5	CaO	2.9	MgO		

Operation of the electric furnace is extraordinarily simple, and the furnace building can be kept almost completely free of gas and dust which is not possible with reverberatory furnace operation.

The furnace has been in full production since June 1949, with a standstill of about three months during the end of 1951 and early 1952 caused by lack of power. There has been no real wear on the brick work. This is in part due to the protective layer of precipitated magnetite on the walls and bottom. Through pyrometers projecting about 250 mm into the brickwork from the outside, one below each electrode, and also a number of pyrometers in the walls, the wear on the brickwork can be controlled. The temperature at the points of measuring seems to have stabilized at about 180° to 200°C.

Table VI gives the most important production data for the years 1950, 1951, and 1952 and Table VII the analyses of matte and slag for 1952.

For normal production in the electric smelting furnace it has been possible to establish statistically

Table VIII. Converter Operation Data for 1950, 1951, and 1952

Data	Quantity*	1950	1951	1952
Blister copper produced	ton	37,150	34,580	37,870
Matte converted	ton	84,200	76,870	81,750
Metal containing scrap treated in converter	ton	9,380	7,510	14,880
Return material including return copper anode scrap	ton	8,285	7,550	6,370
Total copper-bearing material treated	ton	101,865	91,930	103,000
Avg copper content	pct	42.5	43.9	43.0
Quantity of flux	ton	23,000	19,000	19,000
Blowing time per ton of blister copper	min	7.8	7.5	7.7
Amount of air	cu m per min	350	360	360
Amount of gas after Cottrell	cu m per hr	65,000	100,000	85,000
SO_2 content in gas after Cottrell	pct	4.0	3.0	3.5
Converter dust produced	ton	2,000	1,400	1,400
Magnesite brick consumed	kg per ton blister	7.8	6.5	7.0

* All tons are metric tons.

the following relation between the percentage of copper in slag and the percentage of copper in matte respectively and percentages of SiO_2 and CaO:

$$Cu_{slag} = 0.01 \times Cu_{matte} - 0.106 \times SD - 0.036 \times CaO + 0.0017(CaO)^2 + 0.369$$

$$SD = \text{Silica Degree} = \text{molecular ratio} \frac{O_2 \text{ in } SiO_2}{O_2 \text{ in CaO}}$$

When normal operation factors are changed due to process technical reasons the relations in the formula are of course also changed. The formula is used as an aid, taking into consideration current prices of copper, quartz, and lime, to compute the most suitable slag composition to achieve the best economic results.

Since part of the material to be smelted is pyritic and very poor in copper it has been clear from the beginning that this material can be used for slag refining in the electric furnace if it is charged close to the slag tapping end of the furnace. This method when strictly followed has also brought about a lowering of the copper content in the slag with 0.1 to 0.2 pct Cu. This decrease would not be possible if rich and poor material were mixed with each other when charged.

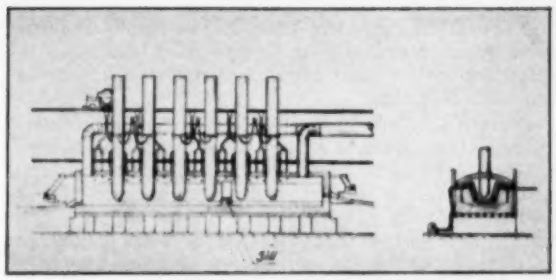


Fig. 6—An air cooled separating wall dividing the furnace into two zones permitted a relatively intensive movement of the bath by means of the electrode current and a remarkable slag refining result was obtained.

With a copper content in the matte of approximately 35 pct a thorough division of the smelting material results in about 0.3 pct Cu in the slag and without such division the slag contains approximately 0.4 to 0.5 pct Cu. Consequently during the months in 1952 when the furnace was not used, an air cooled separating wall dividing the furnace into two zones was built, as shown in Fig. 6. This sill was built as brickwork, resting directly on the furnace bottom, with good sized expansion joints against the furnace walls. It was also weighted with iron weights so that it would not be able to float to the surface. By charging of calcine from arsenic concentrates on the side of the wall closest to the slag tap holes matte was obtained with considerably lower copper content than the matte on the other side of the wall. The current conducted via the electrodes produced a relatively intensive movement of the bath and a most remarkable slag refining result was obtained. It was possible to maintain the copper in the slag at about 0.20 pct while the matte tapped from the furnace had about 35 pct Cu; occasional daily averages of 0.15 pct Cu in the slag and less were also recorded. It seems to be no problem to make the separating wall last for years if the air cooling is satisfactory. The difficulty is in finding suitable material for the cooling tubes which must not be attacked by sulphur or lose their original strength and flexibility through recrystallization.

When the furnace with the sill had been used for about six months the cooling tubes broke, and shortly thereafter the whole separating wall melted. Unfortunately it was necessary to make the cooling tubes of ordinary iron since it had not been possible to obtain heat resistant stainless material in time before production was to be started again. At the earliest opportunity, however, a new separating wall will be built.

Converter Dept.

In 1948, two large converters were purchased from the U.S. measuring 13x30 ft with 52 tuyeres,

Table IX. Analysis of Blister Copper and Converter Slag in 1952

Material	Grams Per Metric Ton		Pct		Pct	
Blister copper	74.0 Au	820.0 Ag	98.57	Cu	0.007	S
			0.01	Bi	0.08	As
Converter slag	—	—	5.4	Cu	0.12	As
			0.29	Co	0.005	Se
			0.36	Ni	1.2	S
			5.25	Zn	27.4	SiO_2
			0.18	Sn	3.4	Al_2O_3
			2.9	Pb	45.4	FeO
			0.002	Bi	0.5	MgO
			0.075	Sb	1.0	CaO

and operation started in 1949. Since then they have been used alternately with one as a spare. The small converters used formerly were removed from the converter aisle.

Certain interesting observations accompanied the change from small to large converters. It appeared that with the same percentages of bismuth and lead in the matte the content of bismuth became higher and that of lead smaller in the blister copper. The reason was easily found. At a total sulphur content of approximately 15 pct, metallic copper starts to separate during the copper blowing period and settles at the bottom of the converter. The distribution ratio for lead and bismuth is such that the larger part of the lead is gathered in the white metal phase (Cu_2S) while the copper phase gathers the bismuth. In the large converter the movement of the bath is so calm that the larger part of the copper during most of the blowing is not stirred up and does not get into close contact with the air. Air contact must continue fairly long in order to remove the bismuth. In the small converters the stirring action was much more intense which made for better refining with reference to bismuth. Consequently when the change was made to the large converters, the bismuth content in the blister copper was increased from a previous 0.005 pct content to approximately 0.10 pct while the lead content decreased from 0.08 pct to 0.02 pct.

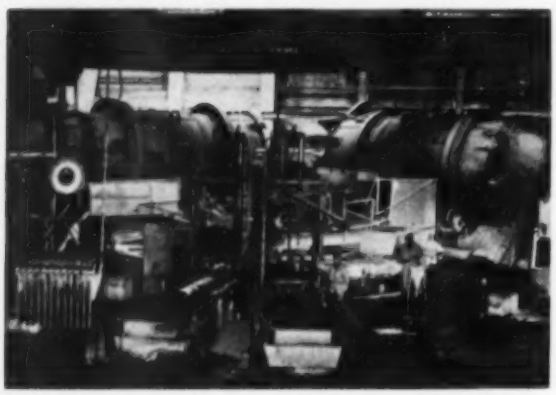


Fig. 7—Speed of the casting wheel can be regulated so that a planned starting and retarding curve can be followed and a smooth surface obtained on the anodes.

The consumption of magnesite brick in the small converters was approximately 40 kilos per metric ton of blister copper produced. In the large converters this consumption has been reduced to approximately 7 kilos per metric ton, a relatively high figure. In order to utilize satisfactorily the advantages offered by large converters, ladles containing 6.5 cu m matte are now used; those used previously contained only 2 cu m.

The converter gases are discharged via bricklined flues with dust pockets into a dust chamber which is also bricklined, and from this chamber the gases can be passed either directly or via a boiler of La Mont type with a 622 sq m heating area to the converter Cottrell. In this boiler, which has been used since March 1951, the gases are cooled to 270°C and the steam production which varies with the converter operation runs between 40 to 100 metric tons daily with 20 kg per sq cm at 380°C. No corrosion of the tubes has been observed.

After the Cottrell the converter gases go to a sulphuric acid plant erected in 1953 with a capacity in its first state of development of 35,000 to 50,000 metric tons per yr of 98 pct sulphuric acid. This plant, constructed in cooperation with and to a large extent delivered by the firm of Chemiebau, Nieder-Marsberg, Germany, has been supplied because of the relatively high arsenic content in the converter gases, with a particularly well-dimensioned washing plant which is followed by a wet Cottrell. The factory is built along the most modern lines and supplied with V_2O_5 contacts. When the converter is not blowing, roaster gases will be used, and in case those should have less than a 3 pct SO_2 content there is an electric gas preheater which can bring up the temperature necessary for the reaction. Tables VIII and IX give data concerning converter operation.

Table X. Average Composition of Anodes in 1952

G Per Metric Ton	Pct			
74.0 Au 820.0 Ag	99.29 Cu	0.26 Ni	0.0018 Sn	0.004 S
	0.014 Sb	0.08 As	0.10 Se	0.010 Bi
	0.046 Pb	0.18 O_2		

Anode Furnace

Modernization in the anode furnace dept. was begun in 1951, resulting in two large anode furnaces of tilting type with capacities of 130 and 145 metric tons respectively being placed in immediate connection to a casting wheel as shown in Fig. 7. The furnaces, which are meant to be used alternatively, are each connected to a very compact aggregate consisting of dust chamber, boiler, and air preheater. The fuel for furnaces is either pulverized coal or oil. The pulverized coal is obtained from a unit pulverizer placed near one of the furnaces. The anode casting wheel, delivered by Maschinenfabrik Augsburg-Nürnberg A.G., Germany and constructed by the same firm in collaboration with the Boliden company, has 22 arms. The machine is constructed in such a manner that all water vapor resulting from the chilling of the anodes and the spraying of the molds is sucked by a strong fan down below the control platform on the casting wheel and through a canal in its center. This arrangement has proved very satisfactory, and even during the coldest days of winter there is no trouble with water vapor. The speed of the casting wheel can be regulated through a special arrangement so that a starting and retarding curve planned beforehand can be followed and a smooth surface of the anodes can be obtained.

Table XI. Average Analysis of Wirebars Cast in 1952

Grams Per Metric Ton	Pct			
0.2 Au 5.0 Ag	<0.001 Ni	0.0306 O_2	<0.0003 Se	
	0.0009 As	<0.0001 Te	0.00046 Pb	
	0.0004 Sb	0.0001 Sn	0.0012 S	
			0.00013 Bi	

In connection to the casting wheel there are two cool boshes and the anodes are lifted from the wheel to one of the boshes with an automatic pneumatic lift and to the other by a manually operated com-

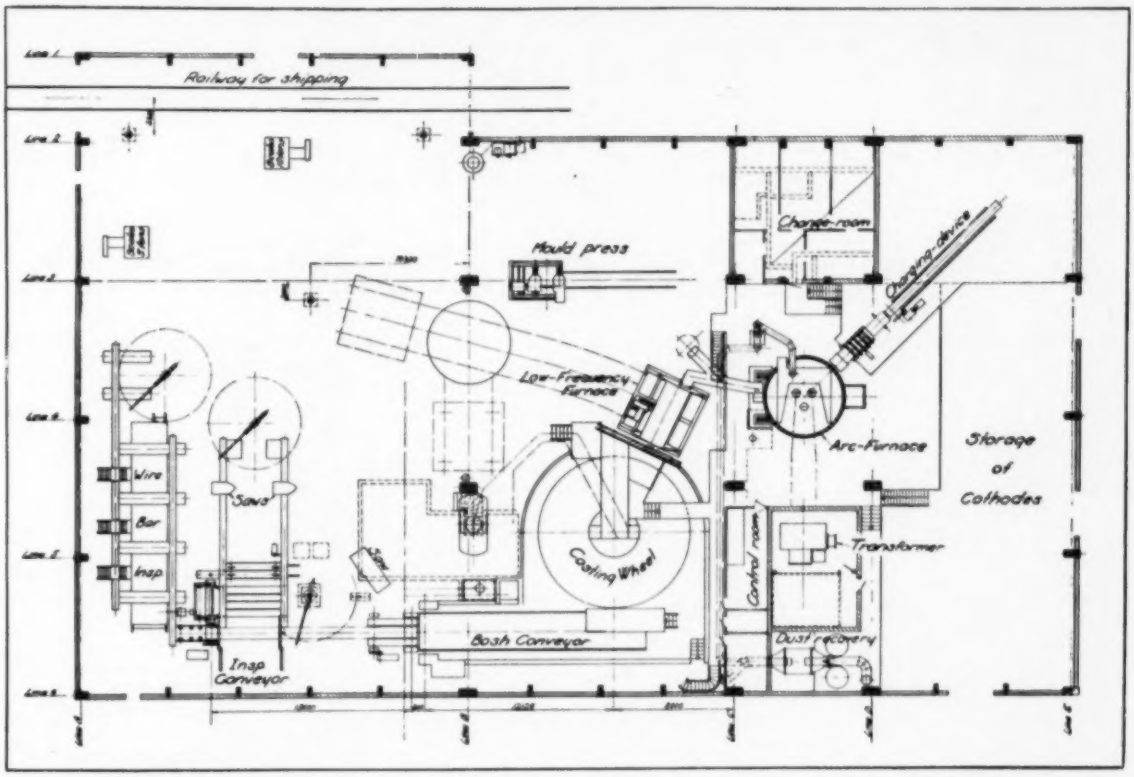


Fig. 8—The cathode furnace department is housed in a building with a floor area of 2490 sq m. The casting wheel is a special type making it possible to cast shapes both horizontally and vertically.

pressed air device which is used as a reserve. In the cold water tank the anodes are suspended from conveyor chains and spaced in the same way as in the electrolytic tankhouse. The bundles of anodes are lifted with a crane and transported by fork trucks into the electrolysis plant. There are a number of stands with a spare supply of anodes in the anode hall. Table X gives the average composition of anodes in 1952.

Cathode Furnace

The original cathode furnace dept. with its two reverberatory furnaces fired with pulverized coal or oil is very obsolete. Here the copper is cast manually after oxidizing and poling, to horizontal wirebars. Productive capacity amounts to about 90 metric tons per day. Table XI shows average analysis of wirebars cast in 1952.

In the old plant it has been possible to make only horizontally cast wirebars while in the new plant completed in 1953 it will be possible to cast practically all sorts of horizontally and vertically cast shapes. The new plant has been housed in a building with a floor area of 2490 sq m divided into four aisles as shown in Fig. 8.

The charging device for the arc furnace is best described as semiautomatic. The cathode packages will be raised on edge by a compressed air device after which another pneumatic device grasps the cathodes one by one at fixed intervals and a machine tosses them so that they slide into the furnace. One man will be detailed at the charging device for supervision and control.

The arc furnace, delivered by Birlec Ltd., England, is a standard construction with certain adjustments demanded by the Boliden company. The furnace is considerably higher than the standard model, while the vertical movements of the electrodes have been diminished, making it possible to use shorter cables and to avoid current losses. The diameter of the furnace shell is 4880 mm and the depth from the bottom to the lower edge of the roof ring is 3140 mm. It is hydraulically tilted. Opposite the spout is a door for slagging and beside it the charging opening. The capacity of the transformer is 6000 kva and the voltage can be regulated as follows: 165, 150, 135, 120, 110, 95, 86.5, 78, 69, and 63.5 v with full effect on the first three tappings. The electrode dimension is 16 in. and the electrode regulating equipment is of the latest ASEA construction which means hydraulic regulation of great sensitivity with very fast reaction, and that the speed of the electrodes can be highly varied from 0 to 0.3 m per sec. In connection with the arc furnace there is a well dimensioned plant for the recovery of copper fumes.

The two low frequency furnaces are comparatively large, each holding 15 metric tons copper. The power input is, however, fairly small, a total of 150 kw on two inductor units. The low frequency furnaces are each placed on a chassis with wheels which means that one furnace can be rapidly replaced by the other in case of accident. These two furnaces were also delivered by Birlec Ltd.

The casting wheel, constructed and in part built by M.H. Treadwell Co. Inc., New York, is of special type, making it possible to cast both horizontally and vertically. In casting vertical shapes, water cooled molds will be used and the heat which the cooling water absorbs can be used either for heating purposes in the cathode furnace building or as feed water to the boilers of the Rönnskär works.

Current Refractory Practice As Applied in Copper Smelting

Present indications are that the highest speeds of copper smelting have not yet been achieved. Although refractories are available which permit reasonable or satisfactory smelting costs, extensive research continues in an effort to develop refractories which will be capable of withstanding even more severe treatment.

by William F. Rochow and Lincoln A. McGill

THE principal furnace equipment of the modern copper smelter which requires careful selection of refractory lining materials might be considered in the same sequence as the plant flow sheet, viz., roasters, reverberatory furnaces, converters, holding furnaces, and anode furnaces.

With progress in ore dressing and concentration and the ability to feed wet concentrates directly to the copper ore melting reverberatory furnace, roasters are not now used by all operating smelters. Consequently, refractories for this application do not now possess the interest that they once enjoyed.

To define the desirable copper roaster refractory in general terms, it should be of alumina-silica type of brick with alumina content of approximately 35 to 40 pct. Workmanship must be the best possible. Reversible thermal expansion should be low and mechanical strength high at moderate temperatures. Density should be high and permeability low. It should be chemically inert to sulphating.

Proper attention should be given to the selection of mortar material for laying roaster brick to protect the joints and to prevent the feed from working into them during times of roaster shutdown. The mortar should possess satisfactory strength, bonding properties, and stability.

Ore Melting Reverberatory Furnace

Of all the furnace equipment now commonly used in copper smelting, the ore melting reverberatory

W. F. ROCHOW is Vice-President, Harbison-Walker Refractories Co., Pittsburgh, and L. A. McGILL is District Sales Mgr., San Francisco.

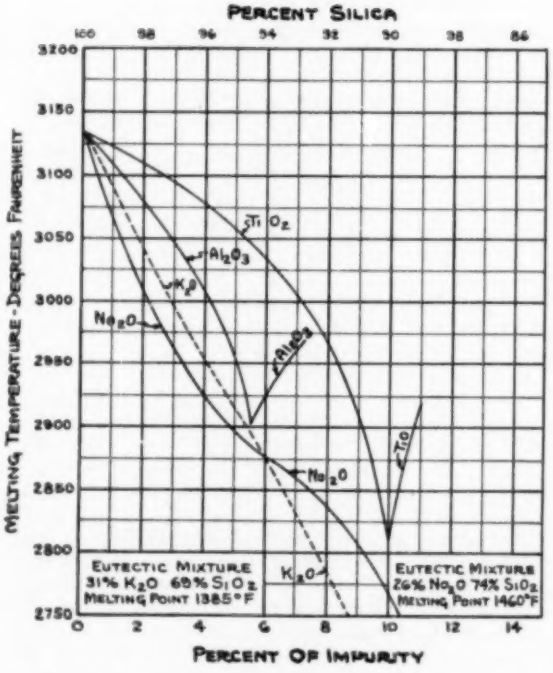


Fig. 1—The melting point of silica is depressed approximately 10°F with 1 pct titania (TiO_2), about 25°F with 1 pct alumina (Al_2O_3), about 45°F with 1 pct potash (K_2O), and about 65°F with 1 pct soda (Na_2O).

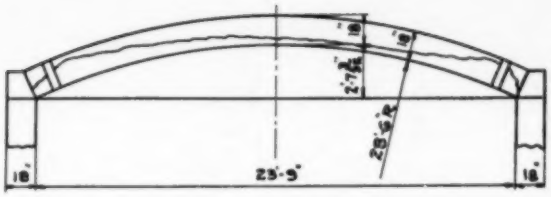


Fig. 2—Typical wear for all-silica roofs of copper reverberatory furnaces is shown in cross section.

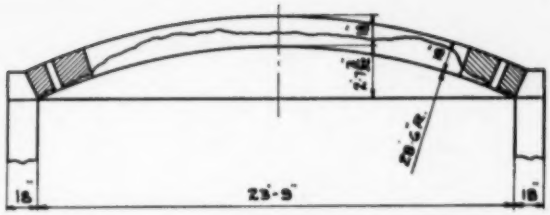


Fig. 3—Typical wear for magnesite-silica roof is shown in cross section (magnesite 4 ft from each skew).

represents the greatest investment. In the application of refractories, substantial progress has been made in the attempt to keep abreast of a progressive increase in the severity of operating conditions brought about by faster rates of smelting.

Considering the basic design and size of this furnace, silica brick lend themselves best to the construction from a mechanical and first-cost point of view. In the high range of temperatures, silica brick are outstanding in their ability to sustain high loads and pressures without deformation. In the temperature range of approximately 1200° to 3000°F their volume remains almost constant. Consequently, in this range, buckstay and tie-rod adjustments are usually unnecessary.

In recent years, much has been accomplished in the improvement and control of the quality of silica brick by beneficiation of the quartzite raw material through washing. The oxides of greatest detriment to silica brick are alumina, titania, and alkalies. The effects of these on the melting point of silica are illustrated by Fig. 1.

Several years ago 1 to 1½ pct of alumina, titania, and alkalies combined were commonly accepted in high quality silica brick. Today, by more careful selection of quartzite in the quarry, which may or may not be subject to other beneficiation, the combined total of these oxides is kept below 1 pct in standard or regular quality silica. Control of the chemical composition of silica brick, in the magnitude mentioned, readily accounts for the ability to operate a furnace at substantially higher temperatures without undue damage to the silica brick.

A noteworthy development in recent years in silica brick has been the commercial production of the super-duty type. With super-duty silica brick, 0.5 pct is set as the maximum allowable combined alumina, titania, and alkalies. As a result, these brick are more refractory and consequently could permit higher operating temperatures of 50° to 100°F than is possible with regular quality product. Likewise, when operating conditions remain the same, service life is usually appreciably increased.

The availability of super-duty silica brick at reasonable first-cost represents an accomplishment of substantial magnitude by the refractory industry. In some cases, it has permitted economical and satisfactory operation where standard silica brick have been inadequate. However, in many reverberatory furnaces, even super-duty silica brick now have relatively short life. With this situation, the remarkable resourcefulness of the smelter operators has been apparent in the development of two methods by means of which they have been able to cope with the conditions satisfactorily.

One method is by daily or semi-weekly gunniting of silica brick with finely ground silica mixed with small percentages of plastic fireclay or bentonite.[1] This material is applied in the form of a spray in thin layers while the furnace is hot. Usually production is not impaired.

Gunniting, as now practiced, resembles a continuous process of maintenance. It is considered economical at many smelters and permits more continuous operation of the furnace. The technique has been developed to the extent that a furnace may be held on the line almost indefinitely if enough gunnite man-hours are expended with judgment and skill. The other method is the successful adaptation of basic refractories in place of silica.

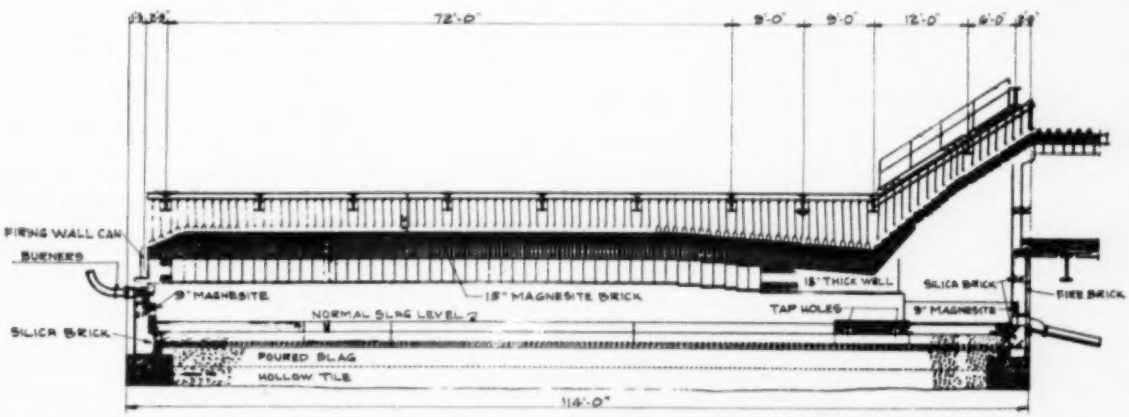


Fig. 4—A longitudinal section of a copper reverberatory furnace showing the suspended basic brick roof construction. Total length of furnace is 114 ft.

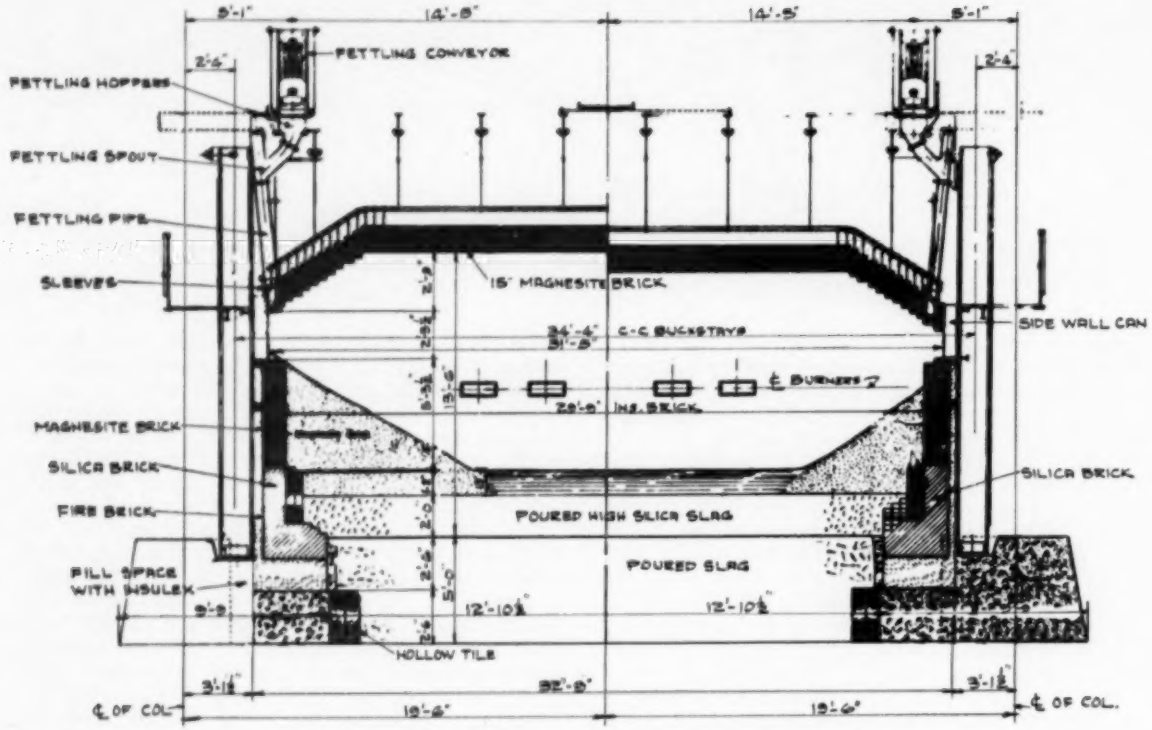


Fig. 5—A cross-section of a copper reverberatory furnace showing the suspended basic brick roof construction. Total width of furnace is 39 ft.

The first successful applications of basic brick to the roofs of copper ore melting reverberatory furnaces consisted of hard-burned magnesite type located in the side charge-hole areas of sprung arches where erosion was most rapid. Silica brick were used in the center sections of these roofs, as illustrated by Figs. 2 and 3.

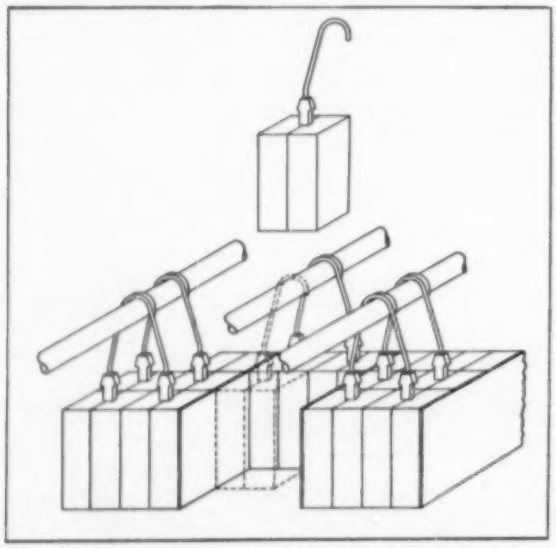


Fig. 6—When the arch requires patching brick are knocked into the furnace in an area of a few feet. New basic brick units are then lowered over the position of the patch and hooked over the supporting pipes.

The combination basic-silica roof construction gave longer service than that of all silica. The adaptability and increased resistance of magnesite brick to the high temperatures and fluxing action of the basic charge were immediately apparent. The magnesite brick charge-hole areas protruded beyond the center silica brick section and the center became the weaker portion of the roof. This was the reverse of the lines of erosion with all silica brick construction. Typical lines of roof erosion with all silica and combination basic-silica sprung arch roofs are also illustrated by Figs. 2 and 3.

No successful technique was developed for patching or replacing the silica center sections of these combination basic-silica roofs. In other words, the life of the roof was governed by the life of the silica center section. When the center section had been eroded to the point of impairing the mechanical stability of the roof, the furnace was shut down and the entire construction was replaced. Recovery and reclamation of the basic brick, as usable bats, was relatively small.

Encouraged by the superior performance of the basic shoulders in sprung roofs, attempts were made to increase the percentage of basic brick in the span. These roofs showed inclination to buckle and spall excessively. They were not considered practical.

From the experiences mentioned, the desirability of adapting basic brick to the entire furnace span was apparent. The problem was how and at what cost. Suspended metal encased chemically bonded magnesite-chrome brick construction has been the answer at several plants.

The first large scale trial of suspended basic brick in the arch of a copper ore melting reverberatory

furnace was in 1932.[8] It was an outstanding success from the beginning. Besides giving longer service and establishing an economy on a refractory cost basis, the suspended basic brick allowed more continuous operation and a faster rate of furnace production. In fact, the credits from the latter accomplishments were possibly of far greater importance than the lower refractory costs derived.

Figs. 4 and 5 illustrate a copper reverberatory furnace with typical suspended basic arch construction for the entire length, including the offtake. Fig. 6 illustrates the basic brick units of the suspended arch, and also illustrates the placement of a unit at time of hot patching. When the arch requires patching, brick are knocked into the furnace in an area of a few feet. New basic brick units are then lowered over the position of the patch and are hooked over the supporting pipes. Only a few square feet are patched at a time. It is not necessary to interrupt or reduce smelting during the operation. Likewise, patching need not follow any specific pattern.

In contemplating the design of the copper ore melting reverberatory furnace of the future, the suspended arch has possibility of permitting furnace sizes and contours that might otherwise be impossible or impractical with sprung arches. For instance, the suspended arch usually recognizes no limitations in regard to size and shape, other than the ability to design exterior steelwork from which it might be supported with safety.

In reverberatory furnace walls, basic brick are now almost universally used at and below the slag line. Above the slag line, silica brick continue to be used at some plants. However, basic brick are gaining ground quite consistently.

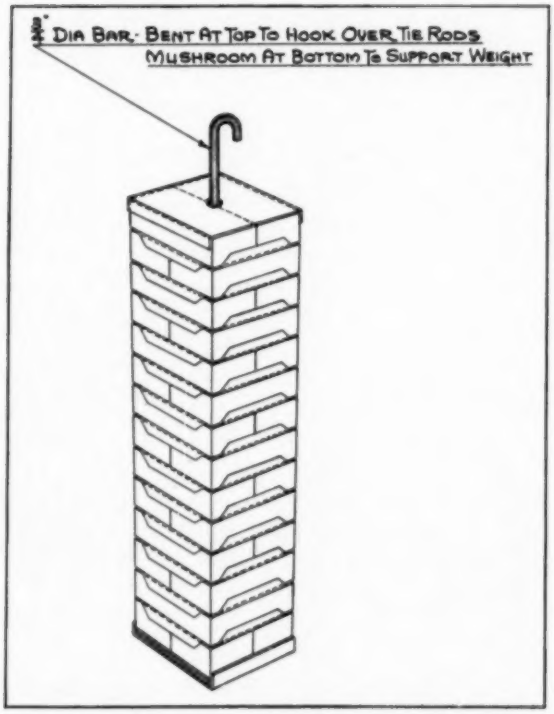


Fig. 7—Basic brick, laid with steel plates, have been used with satisfactory results. A sidewall assembly with steel plates is shown diagrammatically.

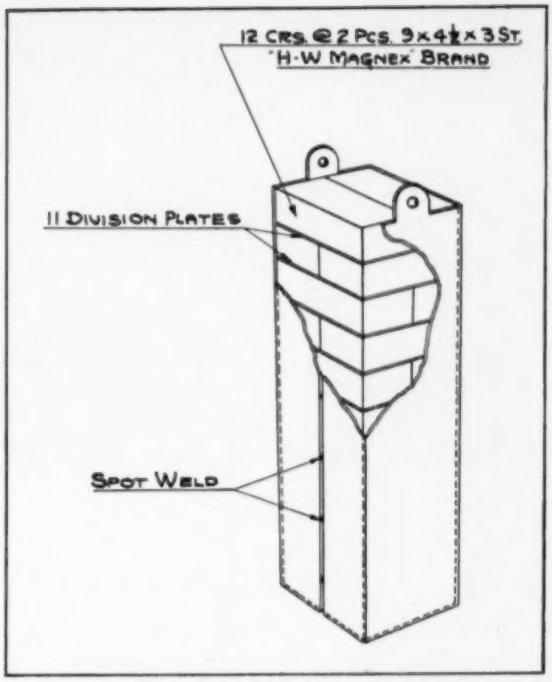


Fig. 8—The sidewall assembly with steel plates and container is another typical construction for basic refractory use in reverberatory furnaces.

The use of water jackets seems to have been the only practical achievement to date that has demonstrated ability to withstand satisfactorily the conditions that result in rapid erosion of refractories at the slag or wash line in deep bath smelting furnaces. In some cases, the wearing face is protected either by a brick sheathing or by a monolithic refractory rammed into a metallic extension of the jacket. In the latter case, the refractory monolith becomes an integral part of the jacket. However, most smelter operators would prefer to avoid the use of water jackets on account of a shortage of water, poor quality of water, or the relatively high heat loss which serves no useful purpose. Likewise, water cooling can represent considerable investment and maintenance cost.

Some furnaces, with basic suspended arches, have sidewall construction that is quite unique but nevertheless very practical and economical. It consists of fabricated sheet steel containers in which monolithic basic material has been rammed and set with a chemical bonding agent. These are commonly referred to as sidewall *cans*. They are suspended from steelwork already existing, or specially provided for the purpose.

Basic brick, laid with steel plates, have also been used for the fillings of these sidewall containers with satisfactory results. However, to date, the metal-encased rammer monolith has the best record for low cost. Typical constructions are illustrated by Figs. 7, 8, and 9.

For offtake and boiler connecting flues, the trend has been from silica and regular grades of fireclay brick to basic, high-alumina and super-duty fireclay types. Suspended arch construction is gaining favor over sprung arches in these positions.

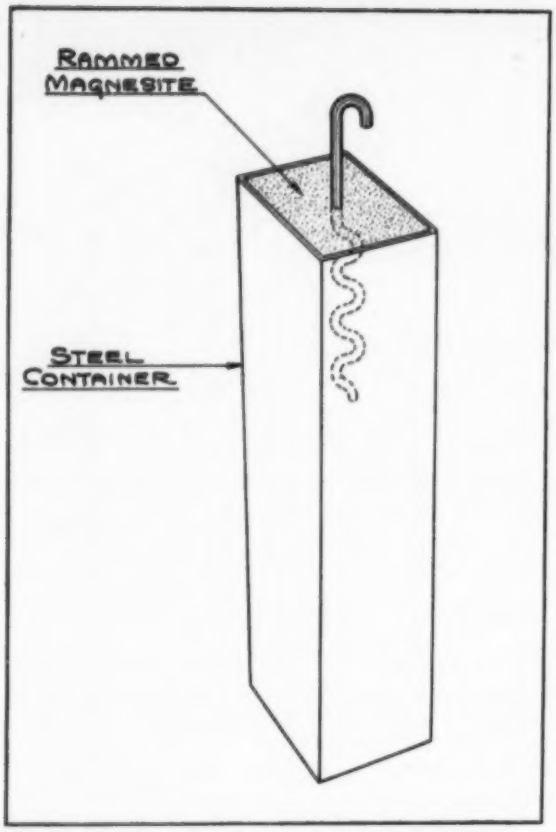


Fig. 9—The metal encased rammed monolith has the best record for low cost. A sidewall metal encased basic refractory is illustrated in this drawing.

For waste heat boiler settings, in which there is not a great amount of black surface in the form of water walls, the higher quality super-duty fireclay brick is now favored over high-duty and intermediate-duty types. In settings designed with substantial water wall area, high-duty fireclay brick have been entirely adequate in most cases.

Converters

Hard-burned magnesite brick were used for the linings of the first basic copper converters. The practice continues to be standard at some smelters. This type of refractory, as now produced, contains about 88 to 90 pct magnesia with excellent strength and density. Other smelters have obtained best results with hard-burned chrome-magnesite brick.

Adequate or correct expansion allowance during installation of converter linings is absolutely necessary for good results with any type of brick. The use of the best obtainable mortar material for laying the brick is also worthwhile. Excellent results are being obtained from the chrome base and high-magnesia types of high temperature cements with cold bonding and setting properties.

Holding Furnaces

Basic brick are universally used for the linings of holding furnaces in all positions with the possible exception of the top. Some smelters use silica, high-duty fireclay, or super-duty fireclay brick for the tops or roofs with satisfactory results.

The basic brick used for maintenance in holding furnaces is usually dictated by the type carried in stock for the converters. That is, they may be either magnesite or chrome-magnesite type of hard-burn process of manufacture. To make this procedure practical, the size or design of the holding furnace must be such that the basic refractory shapes are interchangeable with those of the converters.

With new construction, and likewise for maintenance, it is standard practice at some smelters to use chemically bonded magnesite-chrome brick in holding furnace linings. Chemically bonded brick are serving this application satisfactorily.

Smelter Anode Furnaces

Smelter anode furnaces are referred to herein as distinguished from *refinery anode furnaces*. The charge to the smelter anode furnace consists of molten blister copper. In this equipment, the final stage of smelter pyrometallurgical reduction is accomplished. The product, anodes, is shipped to the refinery for further processing by electrolysis.

While a few of the smelters still retain silica brick in the arches, most of the designs of recent years have been entirely of basic brick. At and below the metal line, hard-burned magnesite brick are used almost universally. However, some of the most recent installations have used hard-burned chrome-magnesite brick. The results have been satisfactory.

With proper installation and reasonably good care, magnesite or chrome-magnesite brick, below the metal line, can be expected to give long service with very little maintenance. Above the metal line, including the roof, hard-burned magnesite and also chrome-magnesite of hard-burned and chemically bonded types were used with satisfactory results.

After service of a year or more, gunnite maintenance of the basic brickwork above the metal line has been necessary or considered advisable. Satisfactory maintenance gunniting is being accomplished with chrome base high temperature cements and also with a mixture of ground and sized dead-burned magnesite. The latter mixture is wet with sodium silicate. The monolithic patch is applied with the *wet* type of gun. With either material, the minimum amount of moisture is used, consistent with the ability to accomplish satisfactory placement.

As with other types of gun or spray applications the technique, with reference to anode furnaces, has been developed to the extent that a furnace may be held on the line almost indefinitely if enough gunniting man-hours are employed with judgment and skill. However, the cost of this practice can develop to substantial proportion. Consequently it is usually a matter of operating judgment to determine when spray maintenance should be terminated and the equipment shut down for basic brick replacement.

It is suggested, whenever possible and practical, that brick types and sizes used in anode furnace linings be interchangeable with those of the converters and anode furnaces.

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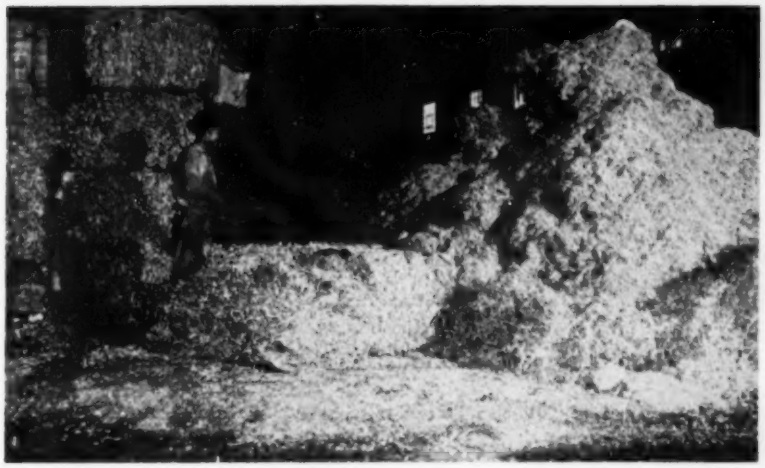

Various sources of scrap including aluminum borings are used in the production of smelters ingot. Aluminum borings are crushed and dried before use.

Direct Reading Spectrograph Gives Accurate Control in Aluminum Production

by Donald L. Colwell and Oldrich Tichy

THE manufacture of aluminum ingot suitable for the making of aluminum castings from aluminum scrap of various kinds has been a feature of the aluminum industry for about 40 years. Originally, aluminum scrap from obsolete pieces of equipment was collected by the dealer, sorted, and run down into ingots with no attention paid to its composition. In those days, aluminum was aluminum, and the producer of aluminum castings did not know much about improving the properties of such castings.

At a later stage the scrap utilized was not only the obsolete scrap from worn out automobiles, washing machines, etc., but included the so-called plant scrap; clippings, rod ends, borings and turnings, etc., from aluminum products of known composition used in various fabricating operations. At the same time, other materials were added to the aluminum scrap to develop smelters ingot of known composition. These other materials might be pure aluminum ingot, or they might be alloying elements, such as copper, silicon, and nickel. The resulting ingot was generally used in the foundry and diecasting industry and became known as secondary ingot.

The term *secondary*, however, had certain connotations not consistent with high quality, and up to the time of World War II, many specifications for high grade material specified that only *virgin* aluminum ingot should be used.

D. L. COLWELL and O. TICHY are Director of Laboratories and Chief Metallurgist respectively, Apex Smelting Co., Cleveland, Ohio. This paper was presented at the AIME Annual Meeting, New York, Feb. 15 to 18, 1954.

It was peculiar that this term applied only to the aluminum industry. The steel industry, for instance, uses large quantities of steel scrap in the production of *primary* steel. The lead and tin industries use lead and tin products from obsolete sources and, in many respects, these are superior to the same alloys made of virgin metals. In the brass industry the term *composition ingot* or *casting ingot* is an accepted quality product to the brass foundrymen even though the origin of this ingot is probably the same automobile or washing machine which produced the scrap for the secondary aluminum ingot.

With the tremendous expansion in the aluminum industry and the concurrent growth in the number of alloys and in the necessary quality control of these alloys, obsolete scrap and plant scrap is used both by the aluminum smelter and by the primary aluminum producer for the production of ingot for the foundryman and diecaster. Specifications for such ingot are becoming more rigid even though all reference to the origin of the ingot or its ancestry is now outdated. Ingot produced from a combination of plant scrap, obsolete scrap, aluminum ingot, and alloying materials has become known as *smelters ingot* and such ingot can be made to the most exacting specifications and for the most exacting uses.

Much of the plant scrap now used in the production of smelters ingot is carefully and accurately segregated, and as such, is simply remelted and cleaned, and provides ingot of a quality equal to that of the original ingot.

When segregation is not carefully and accurately performed, the composition of the resulting ingot

Smelters ingot is produced in open hearth furnaces and can be made to the most exacting specifications and for the most exacting uses.

often has to be adjusted and certain metals either added or diluted so that the resulting smelters ingot will meet the appropriate specifications. It is in this type of control that the direct reading spectrograph has a tremendous application.

Specifications

The American Society for Testing Materials has a specification, B179-53, for aluminum base alloys in ingot form for sand castings, diecastings, and permanent mold castings. In this specification are listed 34 alloys several of which are suited to the production of castings which meet more than one specification. Without going into detail on all 34 alloys, a group of those used only by the diecasting industry is shown in Table I.

In this table the principal alloying elements are copper, silicon, magnesium, manganese, and zinc, and the principal impurity, iron. All of these elements, therefore, are likely to appear in aluminum scrap, and none of them, except magnesium, is successfully removed in the aluminum smelting. In order to meet a specification controlling 8 or 10 elements of greater or less importance, therefore, a careful selection, blending, and control of all types of scrap must be exercised.

An accurate method of chemical analysis is absolutely essential, and the rapidity with which this analysis can be performed not only reduces the time involved in making the alloy but eliminates the necessity for assumptions as to composition and eliminates guesswork in the production of a heat. Such a method is the direct reading spectrograph.

Assay

If all scrap were perfectly segregated, the need for an accurate assay would disappear. Much scrap, however, is mixed, and even when it is supposed to be segregated its composition must be carefully checked to see that it is as it should be. All incoming scrap, therefore, must be accurately sampled and such samples accurately analyzed so that the scrap may be used to its best advantage. Oftentimes, a car of scrap must be sampled and assayed prior to its unloading, because if undue contamination is suspected and confirmed, it is possible that the material would have to be used elsewhere.

It often happens that small lots of obsolete scrap or mixed scrap are put together so as to make a 25 or 30-ton heat which then becomes the raw material for specification grade smelters ingot. Such a heat must also be accurately and rapidly analyzed while it is in the furnace to determine its best disposition.

When a heat of a given alloy is to be made the charge is calculated from the assays mentioned above adding the requisite amounts of the alloying metals and diluting as necessary with pure aluminum or aluminum low in the elements to be diluted. When as many as 10 elements are considered the calculation of such a charge becomes a crossword puzzle type of problem, and the guesswork from this problem can be eliminated only by a knowledge of the exact composition of the several different materials involved in the charge. This composition information must always be at the fingertips of the office where these charges are calculated. This calculation should also consider the type of furnace, the method of charging the various shapes of materials, and the type of ingots into which the heat is to be poured.

Smelting

When an aluminum alloy smelting operation is started in an open hearth furnace a *heel* composed of miscellaneous pigs, heavy castings, or other types of solids is first melted. Although a thorough classification has been made previously on all incoming materials there is always a possibility of unknown contamination. The bath, therefore, is stirred well and sampled at this stage.

This is followed by the charging of materials of smaller size such as borings, turnings, clippings, punchings, etc., and proceeds in the bay of the furnace. As the furnace is charged more samples are taken; the number depending upon the uniformity of the material.

When the heat is nearing completion adjustments are made of major alloying elements such as copper, silicon, manganese, nickel, and zinc. The remainder of the charge is made with known composition materials so that only magnesium adjustments are needed prior to tapping. In order to assure compliance with

Table. I. ASTM Composition Limits for Aluminum Base Alloys in Ingot Form for Die Casting, Pct*

Alloy	Cu	Fe	Si	Mn	Mg	Zn	Ni	Sn	Others (Total
G8A	0.2	0.8	0.3	0.3	7.5 to 8.5	0.1	0.1	0.1	0.2
85C	0.6	1.0	4.5 to 6.0	0.3	0.1	0.3	0.5	0.1	0.2
812A-B	0.6	0.8	11.0 to 13.0	0.3	0.1	0.3	0.5	0.1	0.2
8C84A-B	3.0 to 4.0	1.0	4.5 to 5.5	0.5	0.1	0.9	0.5	0.3	0.5
8C84A-B	3.0 to 4.0	1.0	7.5 to 9.5	0.5	0.1	0.9	0.5	0.3	0.5
8G100A-B	0.6	0.8	9.0 to 10.0	0.3	0.4 to 0.6	0.3	0.5	0.1	0.2

* Percentages shown indicate maximum unless range is given and are selected from ASTM Specification B179-53.

Table II. Analytical History of SC84A Heat of Aluminum

Control	Mg	Zn	Mn	Si	Cu	Fe	Ni	Ti	Cr	Sn	Pb	Zr	Ca
1	0.75	0.86	0.53	4.04	3.05	1.06	0.35	0.04	0.08	0.08	0.09	<0.01	0.001
2	0.62	0.75	0.45	seg	3.70	1.02	0.33	0.03	0.07	0.06	0.09	<0.01	0.005
3	0.45	0.76	0.46	9.40	3.65	0.98	0.34	0.04	0.07	0.07	0.08	<0.01	0.004
4	0.24	0.78	0.44	8.92	3.46	0.97	0.33	0.04	0.07	0.06	0.08	<0.01	0.002
5	0.16	—	—	—	—	—	—	—	—	—	—	—	—
6	0.06	—	—	—	—	—	—	—	—	—	—	—	—
Final													
1	0.06	0.75	0.43	8.98	3.48	0.96	0.32	0.04	0.07	0.08	0.08	<0.01	0.001
2	0.05	0.78	0.45	8.87	3.40	0.97	0.34	0.04	0.08	0.07	0.07	<0.01	0.001
3	0.06	0.76	0.44	8.92	3.50	0.95	0.34	0.03	0.07	0.08	0.09	<0.01	0.001

chemical specifications a final control sample is taken while preparations are made for pouring. As a last check, final samples are taken while the pouring proceeds; these are carefully spaced at the beginning, middle, and end of the pour.

Direct Reading Spectrograph

The analytical problems have directly followed the trend of the industry. As the need for accurate composition increased, so did the necessity for more rapid and more accurate analysis increase. In the past, rapid and not too accurate wet chemical methods were used exclusively for the analysis of copper, iron, silicon, manganese, zinc, and sometimes nickel. Only estimates were made of some minor elements to assure the absence of excessive amounts.

With the advent of the spectrograph some elements were analyzed spectrographically and some by wet methods, and as the need arose for more rapid analysis to reduce furnace time, more determinations were delegated to the spectrograph.

Several years ago, when the first direct reading spectrographs were being developed, it was hoped that with this type of instrument the analytical time would be reduced considerably. Such proved to be the case as the time of wet analysis was about 3 hr, that of spectrographic analysis about 1 hr, while that of the direct reading spectrograph is about 10 min. Because of this short analytical time any discrepancy in composition is immediately brought to the attention of the production dept. and is remedied as the charging proceeds. Another advantage is that more frequent sampling can be done when necessary and instead of composite samples being used, different parts of the bath can be analyzed separately, thereby informing the production dept. if any segregation is present.

Standards

The spectrographic methods of analysis, whether they be made photographically or by direct reading, depend on comparison with known standard samples. In order to assure the greatest accuracy, considerable time is devoted to producing the best possible standards. Each alloy is covered by three samples covering a range of all the alloying elements.

Much care is taken in alloying the metal and in pouring these standard samples. They are then scanned for uniformity on the direct reading spectrograph and are finally analyzed by wet umpire methods which are recognized as the best available. Working curves are made from these standards for use in analyzing the unknown samples. These curves are continually checked with the standard samples to insure that all elements are recording properly. After any adjustments the standards are again used to check the curves, and by continual checks and rechecks, the analyses are kept accurate.

Typical Heat History

Table II shows a typical analytical history of a heat of SC84A or 39 aluminum. Control sample on line one gives a typical analysis of remelt ingot. The silicon and copper contents are low while the magnesium is high. Attention is called to the segregated silicon in the second control sample which notifies the shop of insufficient stirring after the addition of this element. Control samples on lines three and four follow the progress of further charging of material. Note the reduction of iron content by dilution. At the end of charging only magnesium reduction is necessary. When this is accomplished the heat is tapped and three final samples are taken to insure uniformity of analysis throughout the heat. The average of these three is the composition of the heat.

Control Cost

While the direct reading spectrograph is the ideal instrument for close control of quality as outlined above, its cost is not negligible. The instrument must be operated around the clock, and as an installation can cost as much as $100,000, the overhead becomes an appreciable item. Some typical results, however, might be found interesting.

To produce 120 heats a month, about 1600 complete analyses are made. About one third of these are on the incoming material, and two thirds are preliminary samples and final samples of the heats in the furnace. This is done with a payroll of about $2400 per month. The direct labor cost, therefore, of this type of analytical control is $20.00 per heat, and the total cost about $40.00 to $60.00 per heat. With just half as many heats, the above cost would be doubled, whereas the same equipment could turn out the analytical work for about twice as many heats if the volume were there. A cost, therefore, of $50.00 per heat is probably typical. The principal advantages gained are two: First, a saving in furnace time which is tremendous but hard to calculate, and second, an improvement in uniformity and in quality that could not be obtained by any other type of analysis.

Control Advantages

The accuracy and speed of this equipment are much superior to those of the previous analytical methods where personal techniques are very important. This is reflected in the intangible aspect of much greater harmony and efficiency between laboratory and plant personnel, and can be as important as the tangible aspect of much greater uniformity of product and practical elimination of rejected heats. The quality of aluminum smelters ingot, therefore, can be accurately maintained and commercially obtained by the intelligent use of the direct reading spectrograph.

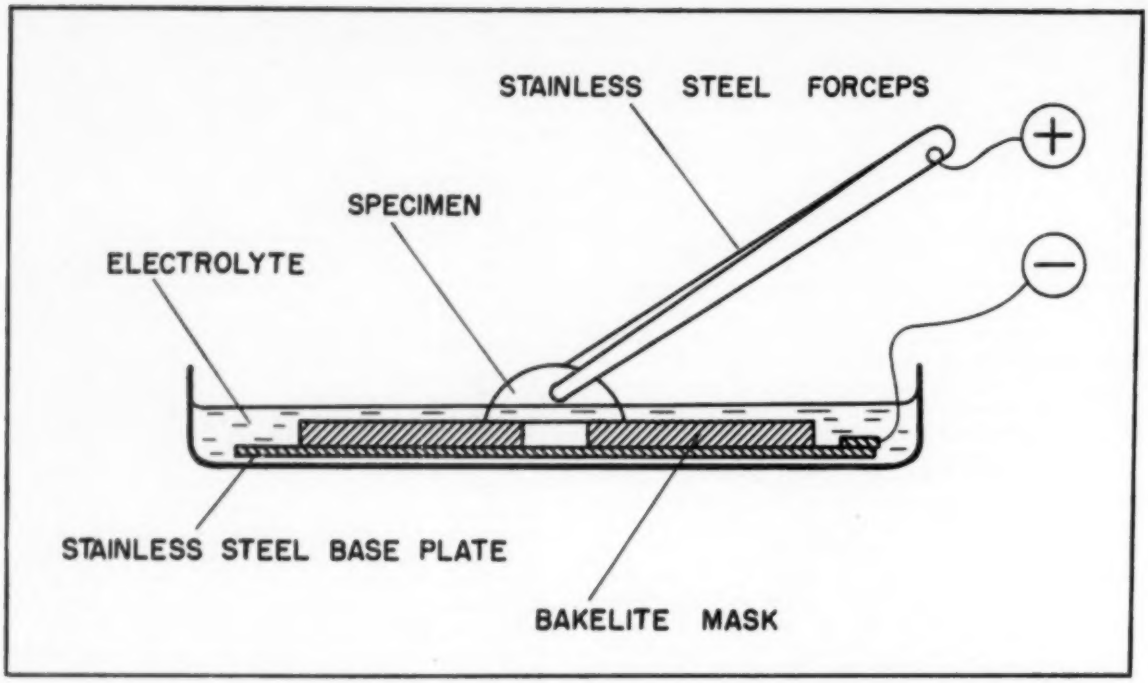

Fig. 1—The device used for cumulative electrolytic stain-etching is shown diagrammatically. Current densities are determined by the thickness of the Bakelite mask and the size of the opening of the mask.

Phases in Titanium Alloys Identified by Cumulative Etching

by Elmars Ence and Harold Margolin

IN the course of phase diagram work conducted at New York University, it was found that metallographic identification of phases by the use of ordinary etching techniques was quite often uncertain. To aid in distinguishing between phases, heat-tinting and electrolytic stain-etching techniques were developed.[1, 2] The use of both heat-tinting and stain-etching for phase identification is based on a differential rate of oxidation of microconstituents.

Heat-tinting has been used principally for developing color differences between alpha, retained beta, and carbides. For heat-tinting, specimens approximately 3/16x3/16x3/8-in. are electropolished, etched, carefully rinsed in water and alcohol, and dried. The specimens are then placed in a muffle furnace on a metal plate maintained at 600°C and allowed to heat for 60 sec. Under these conditions the microstructures develop the following colors:

A—Retained Beta–deep violet to bright blue.
B—Alpha (isothermal or equiaxed)–dull yellow to golden yellow.
C—Martensite–as alpha but with some violet coloring due to the presence of retained beta in intimate mixture with martensite.
D—Carbides–brilliant bright blue or yellowish.

Repeated treatments on the same specimen may cause transformation of retained beta, in which case beta will appear yellowish.

At temperatures below 500°C orientation effects were frequently encountered and often colors were not constant. These difficulties did not occur in the region from 500° to 600°C, and color distinctions were more pronounced as heat-tinting temperatures approach 600°C. On the whole, heat-tinting is a less convenient technique than stain-etching. It is

E. ENCE and H. MARGOLIN are Research Associates, College of Engineering, New York University, New York, N. Y.

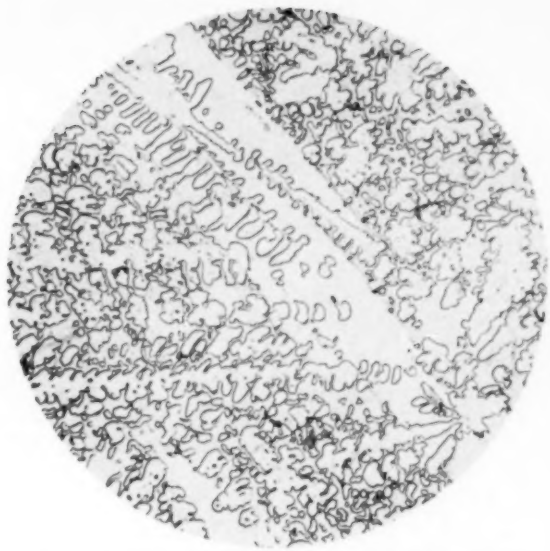

Fig. 2—The microstructure of an as-cast Ti-54.5 Mn alloy etched with HF + glycerine (1:1:10), magnified 350X shows two or three phases. The microconstituents are: $TiMn_{(1-2)}$, ϵ, and β-titanium.

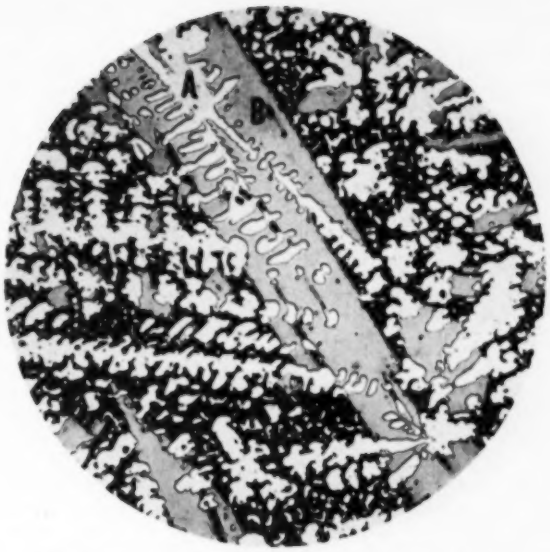

Fig. 3—The same alloy as Fig. 2 electrolytically stained (*HG* electrolyte, 5x0.3 sec at 125v), magnified 350X, shows: A—$TiMn_{(1-2)}$, actually bluish green, B—ϵ actually violet, and black β-titanium actually purplish red.

slower and the structure is subject to modification during the time necessary for developing oxidation colors. For these reasons, heat-tinting has been limited to the simple structures described.

Stain-Etching

The work in electrolytic stain-etching can be classified under swab-etching and cumulative etching which refer to the procedure used in developing the stain. In swab-etching[1, 2] the specimen, after polishing (mechanical or electrolytic) and etching with a standard reagent, is placed on a stainless steel anode. The tip of a stainless steel rod, the cathode, covered by a small cotton wad, is wetted with the 10 pct NaCN solution and the circuit is closed by touching the pre-etched surface of the specimen with the wad. The distance between the specimen and the cathode is determined by the thickness of the cotton wad, and this is kept less than ⅛ in. Etching times of 60 sec at 8 to 10 v and 0.14 amps were used.

When electrolytically etched, the specimen surface appears violet or blue. Under the microscope carbides are yellow or orange-yellow, while alpha and transformed or retained beta appear violet or blue. This etchant is also applicable for distinguishing between the boride phases Ti_2B, TiB, and TiB_2.[3]

Often it was found that the NaCN etch at 8 to 10 v could not produce distinctive differences between some phases, particularly those of the Ti-Mn and Ti-Mn-Fe systems. Indications were obtained that at higher voltages better phase differentation could be effected. However, severe scoring of the surface took place as a result of sparking, which occurred when the swab was moved, and frequently reaction products obscured the microstructure. In addition, swabbing prevented accurate control of current densities and uniform oxidation of the surface.

Cumulative etching was developed to alleviate the difficulties encountered with swab-etching. With this method the electrolytes which have proved most widely applicable for stain-etching purposes are identified as *H-etch* and *HG-etch*. H-etch consists of: 5 g oxalic acid, 5 g citric acid, 5 ml orthophosphoric acid 85 pct, 10 ml lactic acid 85 pct, 35 ml water, and 60 ml ethanol 95 pct.

HG-etch is made by diluting H-etch with an equal volume of glycerine. Dilution with glycerine increases the total time required for etching and thus increases the control over stain-etching. HG-etch is used in those cases where sufficient contrast cannot be obtained with the faster H-etch. It should be noted that the colors obtained with HG-etch are not the same as those achieved with H-etch.

Cumulative Etching Procedure

In the cumulative etching procedure, the polished and etched (standard reagent) specimen is placed as shown in Fig. 1 and the circuit is closed by an external switch. Since the specimen is rapidly

Table I. Change of Specimen Surface and Microconstituent Colors with Staining Time for Ti-30Fe-6Mn Alloy Stained with H-Etch

Total Staining Cycles	Surface Appearance	Ti-Fe	Microconstituents Ti-Mn-Fe Compound	Retained β-Ti
2x0.3sec-125v	violet	light blue	pale violet	violet
3x0.3sec-125v	blue	white	gray blue	blue
5x0.3sec-125v	orchid	orchid	pink	yellow
7x0.3sec-125v	violet blue	aquamarine	gray pink	purple
10x0.3sec-125v	greenish blue	light green	deep aquamarine	purple

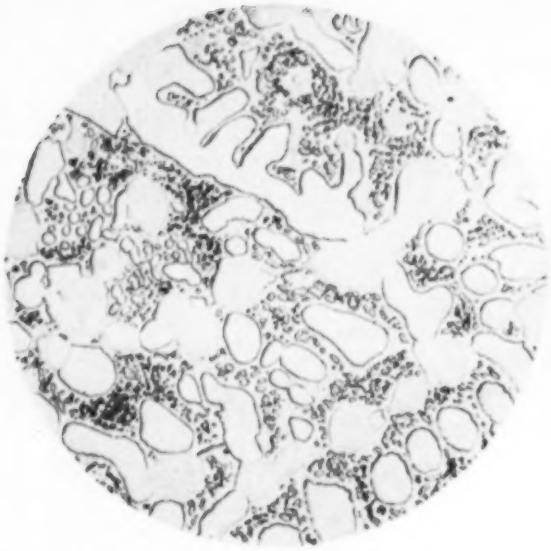

Fig. 4—Microstructure of Ti-30Fe-6Mn alloy (264 hr — 700°C/W. Q.) etched with HF + HNO_3 + glycerine (1:1:2 magnified 350X shows the microconstituents TiFe, Ti-Fe-Mn, and β-titanium.

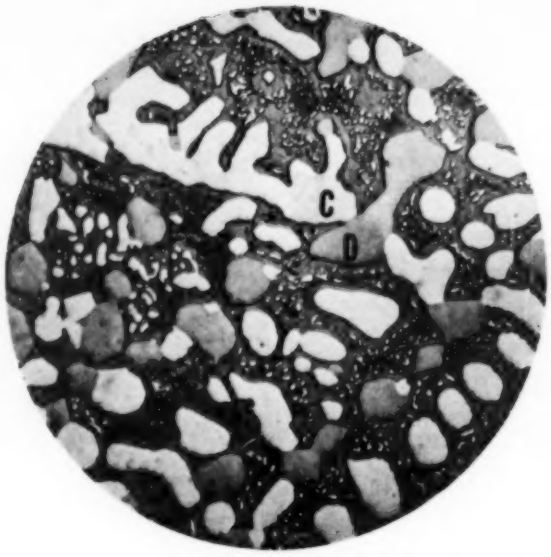

Fig. 5—Same alloy as Fig. 3 electrolytically stained (*HG* electrolyte, 7x0.3 sec at 125 v) magnified 350X shows: C—TiFe actually bluish green, D—Ti-Fe-Mn actually violet, and dark β-titanium actually orange.

heated and interfering gas bubbles are formed, the time for one etching cycle must be kept to a fraction of a second. To obtain a properly etched surface, the circuit is opened and closed 5 to 10 times.

Current densities are determined by the thickness of the Bakelite mask (2 to 6 mm) and the size of the opening in the mask. Convenient areas of openings are 25, 50, and 75 sq mm. Applied voltages in the range from 20 to 130 v are most frequently used, and for best differentiation of phases, 100 to 130 v are employed.

With cumulative etching, the surface of the specimen gradually changes color. At the outset, it appears yellow, and with additional etching the color changes to brown, violet, and blue. With further increase in etching time the color cycle is repeated with some modification. Generally, the best distinction between phases occurs when the blue color appears for the second time. The etching time for appearance of the blue color will vary with composition and type of alloying constituents. An illustration of the changes in color of the microconstituents with increasing etching time for a Ti-30Fe-6Mn alloy is shown in Table I.

It has been pointed out that a part of the procedure is to etch the polished surface with standard etchants prior to stain-etching. This is not necessary, but is done to give a sharper outline of constituents and to improve the general appearance of the microstructure. When pre-etching is not done, the colors obtained are consistent, but are different from those developed by pre-etching. In some instances where pre-etching is not desirable but where the colors associated with pre-etching are wanted, it is possible to obtain the pre-etched colors by first making the specimen cathodic in the H-etch electrolyte for a short time.

To illustrate the effect of cumulative stain-etching in producing contrast between phases, two sets of microstructures are shown in Figs. 2, 3, 4, and 5. In each set the same area is shown as etched by standard etchants and by stain-etching. The colors of the microconstituents in the figures do not coincide with those of Table I because HG-etch was used for these illustrations.

Figs. 2 and 3 show a microstructure about which some controversy exists in the literature.[4, 5] Fig. 2 may be said to show two or three phases. From Fig. 3 it can be seen that three phases exist. This is a microstructure of an as-cast Ti-54.5 Mn alloy. The gray phase (actually violet) is ϵ. The white phase (actually bluish green) is a hexagonal phase with a lower manganese content than the ϵ phase, and the dark matrix (actually purplish red) is retained beta.

Figs. 4 and 5 are microstructures of a Ti-30Fe-6Mn alloy heat treated at 700°C. The white phase (actually bluish green) of Fig. 5 is TiFe, the gray phase (actually violet) is a ternary Ti-Fe-Mn compound and the matrix (actually orange) is retained beta.

Acknowledgment

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Application of the ARL Quantometer to Production Control In a Steel Mill

by H. C. Brown

SINCE 1934 the steel industry has been utilizing the spectrograph for supplementing wet chemical analysis in the production control of electric and open hearth furnaces. This means of control made great strides during the war years because of the general acceptance of the spectrograph and the increased emphasis that was placed on rapid control methods. However, in the post war era, with the demand still on increased production, it became apparent that a still more rapid and economical means of production control was needed. Since the spectrograph had been used mostly in the analysis of low alloying and residual elements, it also became apparent that equipment was needed to extend the spectrographic technique to the analysis of the high alloying elements in stainless steel.

For these reasons, companies manufacturing spectrographic equipment were prompted to start development work on direct reading instruments. In June 1949, the Applied Research Laboratories of Glendale, Calif., announced that a direct method of spectrochemical analysis for stainless type steels had been developed. This paper will describe the use of the Applied Research Laboratories Production Control Quantometer in the quantitative control of stainless, silicon, and plain carbon steels being made at the Butler Pennsylvania Div. of the Armco Steel Corp.

The Armco Butler Div. has one 70-ton electric furnace and six 150-ton open hearth furnaces. The electric furnace is employed in the making of all types of stainless steel and the open hearth furnaces are used for the production of silicon, wheel, and plain carbon steels. The ARL quantometer was purchased primarily for the purpose of controlling the steelmaking in the electric furnace, but its use has been extended for the analysis of final tests (ladle tests) on a number of different types of stainless, silicon, and plain carbon steels. Because of this additional work by the quantometer, substantial savings in manpower and time have been realized by the laboratory. In the analysis of a set of preliminary tests from the stainless steel furnace, approximately 40 min in laboratory time are saved due to quantometric analyses. Despite the fact that more specialty grades of stainless steel are being made in the electric furnace, the average tons per hour have been increased since the quantometer was put into operation. Specialty grades require more furnace time than regular commodity grades of stainless steel.

The installation of the ARL production control quantometer was completed on March 13, 1952. By May 1, 1952, the instrument was calibrated for nickel, chromium, manganese, silicon, and molybdenum, which are the elements necessary for the production control of the stainless steel furnace. Within the following month, training of personnel on the quantometer was achieved and a study of the accuracy of the instrument showed that the results obtained were sufficiently accurate for control purposes. Therefore, on June 11, 1952, the quantometer was placed on production control for all types of stainless steels. Starting September 11, 1952, the instrument was gradually placed on ladle analysis (final tests) as the analytical curves were refined and additional curves were drawn.

The quantometer has been relatively free of breakdowns since placing it on production control. The samples from only one stainless steel heat have had to be analyzed by wet chemistry because of instrument trouble. The previously existing heat-time record was also bettered by 15 min on a commodity grade of 18-8 stainless steel.

Scope of Control

In general, the quantometer determines all elements necessary for the production control of all types of stainless steel heats and for the ladle analysis of various types of stainless steel heats. It is also used in reporting final results for silicon, manganese, chromium, nickel, molybdenum, tin, copper, and aluminum on all silicon steel grades and manganese, chromium, nickel, molybdenum, tin, and copper on several plain carbon steel grades.

Table I shows the elements and the concentration ranges of these elements in the various types of stainless, silicon, and plain carbon steel that are determined on the quantometer. A study of the results obtained on ladle test samples of stainless steel types 410, 430, 430 Ti, 446, 301, 302, 304, 304L, 305, and 17-7 PH will be discussed. Also included in the study are the results obtained on ladle test samples of a number of silicon steels.

Apparatus

In order to take full advantage of the potentials of the production control quantometer, the unit has been placed in an air-conditioned room with relative humidity control. The temperature is maintained at 73°±2°F and the humidity at 45±5 pct. The air conditioning serves as a precaution to minimize the amount of adjustment and calibration needed during operation. It also reduces contaminating fumes and dust and thereby lessens the necessity for maintenance on the equipment.

The quantometer is composed of three units: the high precision multisource unit, the 1.5 meter vertical spectrometer, and the console. The source unit supplies excitation conditions varying from spark-like discharges to arc-like discharges. The voltage to the source unit is supplied by a motor-generator

H. C. BROWN is associated with Armco Steel Corp., Butler Div., Butler, Pa.

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The Transactions papers appearing in the Journal of Metals during 1954 will subsequently appear in AIME Transactions Volume 200, and may be permanently referenced to that volume.

Table I. Concentration Ranges of Elements Determined by Quantometer for Various Types of Steel

Element	Type of Steel	Concentration Range, Pct
Ni	Stainless type 410, 430, 430 Ti, 446	0.05 – 1.25
Ni	Stainless type 301, 302, 304, 304 L, 305, 17-7 PH	4.00 –11.00
Ni	Silicon	0.005– 0.50
Ni	Plain carbon	0.005– 1.00
Cr	Stainless type 410, 430, 430 Ti, 446	9.00 –28.00
Cr	Stainless type 301, 302, 304, 304 L, 305, 17-7 PH, 321, 316	10.00 –20.00
Cr	Silicon	0.005– 0.50
Cr	Plain carbon	0.005– 0.70
Mn	Stainless type 410, 430, 430 Ti, 446	0.05 – 1.00
Mn	Stainless type 301, 302, 304, 304 L, 305, 17-7 PH	0.05 – 1.50
Mn	Stainless type 309, 321, 316, 310 S, 310 S Ti	0.05 – 2.25
Mn	Silicon	0.005– 0.60
Mn	Plain carbon	0.10 – 0.70
Si	Stainless type 410, 430, 430 Ti, 301, 302, 304, 446, 304 L, 305, 17-7 PH, 309, 321, 316, 310 S, 310 S Ti	0.05 – 1.25
Si	Silicon	0.90 – 5.25
Cu	Stainless type 410, 430,430 Ti, 301, 302, 304, 304 L, 305, 17-7 PH, 309, 321, 316, 310 S, 310 S Ti, 446	0.10 – 0.60
Cu	Silicon	0.05 – 0.50
Cu	Plain carbon	0.05 – 0.50
Ti	Stainless type 430	0.02 – 0.25
Mo	Stainless type 316	1.50 – 3.00
Mo	Stainless type 410, 430, 430 Ti, 301, 302, 304, 446, 304 L, 305, 17-7 PH, 309, 321, 316, 310 S, 310 S Ti	0.005– 0.50
Mo	Silicon	0.003– 0.050
Mo	Plain carbon	0.003– 0.060
Al	Stainless type 17-7 PH	0.80 – 1.40
Al	Silicon	0.003– 0.35
Sn	Stainless type 410, 430, 430 Ti, 301, 302, 304, 304 L, 305, 17-7 PH, 309, 321, 316, 310 S, 310 S Ti, 446	0.005– 0.10
Sn	Silicon	0.005– 0.10
Sn	Plain carbon	0.005– 0.10
Pb	Stainless type 410, 430, 430 Ti, 301, 302, 304, 304 L, 305, 17-7 PH, 309, 321, 316, 310 S, 310 S Ti, 446	0.001– 0.015

set and is regulated by a Westinghouse SRA-3 Silverstat regulator.

The spectrometer is constructed of a box-type aluminum weldment, vertically mounted, and contains the diffraction grating, photomultiplier tubes, mirrors, and slit assemblies. The diffraction grating installed in this instrument is ruled 24,400 lines per inch. The instrument has a dispersion of 6.95Å per mm and a resolution of 0.5Å when using a 0.006 in. slit. The primary or entrance slit is 50 microns wide and the secondary or exit slit for the element and internal standard lines are 150 microns wide. The secondary slits are semifixed and can be adjusted so that the maximum amount of light will fall on the mirrors which in turn directs the light onto the photomultiplier tubes.

The console houses all the necessary high and low voltage power supplies, the condensers, the electrometer amplifier, the calibration sensitivity controls for all element ranges, the phototube voltage controls, the switching circuits, and a recording system. The condensers store the integrated phototube output, the amplifier measures the voltage on the condensers, and the recording system records the amplifier response. The recording system gives an inked record of all analyses. The calibration sensitivity controls are used to standardize the recorder scale for the particular ranges desired.

A calculating machine is used to speed up calculations and is especially desirable for preliminary analyses where time is an important factor. A Campbell model 406 cut-off machine and two belt surfacers are used for sample preparation.

Sample Preparation

It has been found that sampling is one of the most important factors for accurate quantometric analysis. Since the spectrometer unit is equipped with a Petry stand, the point-to-plane technique of analysis is employed. A flat sample is used as the positive electrode while a 120° carbon is used as the negative electrode. A gap distance of 3 mm separates the two electrodes.

The sampling procedure being used is as follows: the furnace or ladle sample is poured into a round cast iron mold, 2¾ in. deep and 2½ in. in diameter at the top and 2¼ in. in diameter at the bottom. This sample is water-quenched and a slice approximately ⅜ in. thick is cut from the bottom of the sample with the automatic cut-off machine. The final surface of the sample is prepared on a belt surfacer with a 180 grit aluminum oxide belt. The polished sample is then sparked approximately ½ in. in from the outside edge. This location is within the perpendicular dendritic field where it is believed that better reproducibility should be obtained as shown by previous work in the Armco Research Laboratories.

Excitation and Line Pairs Used

Two discharge conditions from the multisource unit have been chosen for the analysis of stainless, silicon, and plain carbon steels. A spark-like discharge with multisource settings of 10 microfarads capacitance, 90 microhenries inductance, and 5 ohms resistance is used for the analysis of the alloying elements. An arc-like discharge with multisource settings of 40 microfarads capacitance, 550 microhenries inductance, and 25 ohms resistance is used for the analysis of the residual elements.

The line pairs with the percentage range for which they can be calibrated are shown in Table II.

Standards

All quantometric analyses are based on standard samples. Two standards, in the case of stainless steel analyses, are run before each unknown and the average deviation of the standards from the chemical values is applied to the unknown samples. The standard samples were chosen in the following manner: test samples covering a broad percentage range for all elements were taken from a large number of production heats. The samples which proved to be most reproducible on the quantometer were then carefully chemically analyzed by the Armco Research Laboratories and the chemical laboratory of the Armco Butler Div. These samples were then used to construct standard analytical curves and the samples which gave readings that fell on or near the curve were used as permanent standards. In some cases laboratory heats were made for standards, especially the calibration standards.

It might be suggested here that a new quantometric laboratory obtain a set of their own standards from production heats made in their own plant. It is evident that the metallurgical properties of samples can vary due to variations in methods of sampling. These characteristics must be standardized.

Calculations

General operation of the quantometer will not be discussed since a regular procedure can be formulated and standardized by each laboratory. However,

methods of calculation in determining the analysis of a stainless steel preliminary or ladle sample will be explained. First, two standard samples which have been accurately analyzed by wet chemical methods are sparked at least four times and allowed to record each time on the console recorder. The average chart reading for each element of the standard is converted to percentages from a standard working curve. The corrections, which will be applied later to the preliminary or ladle sample, are then determined by comparison with the previously analyzed chemical results. The preliminary or ladle sample is then run at a predetermined duodial setting. The duodial is a control placed on the sensitivity knob of the internal standard iron channel which regulates the exposure time to give a predetermined integrated intensity of the standard iron reference line. After the preliminary or ladle sample has been sparked once and allowed to record, the chart reading of each element to be determined is converted to percentages by reading the calibration curve for that particular element. The percentages of the elements are then added along with the iron percentage which has been introduced in the instrument by the duodial control knob. If the total of these percentages is between 98 and 102 pct, the sample is then run at least two times on a preliminary test and four times on a ladle test, and the average chart reading of the individual runs is taken for each element. The new chart readings are converted to percentages and again added along with the iron percentage. The sum of the percentages is then divided into 100, and the factor obtained is applied to the percentage of each element. The sum of the calculated percentages should equal 100. The corrections, if any, as indicated by the standards that were previously run are now applied to the calculated percentages of each element to give the final result.

In the first calculation of the unknown, if the total percentage does not fall between 98 and 102 a new duodial setting is placed in the instrument, thus correcting the iron percentage and making the total percentage fall within the specified limits. A curve, duodial reading vs percentage iron, has been determined so that a new duodial setting can be chosen quickly and set in the instrument. Since the standards have been run a number of times, duodial settings have been established and the same duodial setting is used each time the standard is run.

Table II. Element and Internal Standard Iron Lines Used in Analysis of Steels

Element Line	Iron Line	Concentration Range, Pct
Stainless Steels		
2270.2 Ni	2714.4 Fe	0.05 – 2.0
2316.0 Ni	2714.4 Fe	2.0 –10.0
2316.0 Ni	2714.4 Fe	9.0 –30.0
2677.2 Cr	2714.4 Fe	0.5 – 2.0
2860.9 Cr	2714.4 Fe	2.0 –12.0
2860.9 Cr	2714.4 Fe	10.0 –30.0
2949.2 x 2 Mn	2714.4 Fe	0.05 – 2.5
2516.1 Si	2714.4 Fe	0.1 – 2.0
2516.1 Si	2714.4 Fe	1.0 – 5.0
3372.8 x 2 Ti	2714.4 Fe	0.1 – 1.0
3195.0 x 2 Cb	2714.4 Fe	0.2 – 2.0
2816.2 x 2 Mo	2714.4 Fe	1.0 – 5.0
3170.3 Mo	4404.7 Fe	0.005– 0.5
3961.5 Al	2714.4 Fe	0.1 – 1.5
3274.0 x 2 Cu	2714.4 Fe	0.1 – 1.0
2242.6 Cu	2714.4 Fe	0.5 – 3.0
2833.1 x 2 Pb	4404.7 Fe	0.005– 0.2
3262.3 Sn	4404.7 Fe	0.005– 0.2
4008.8 W	4404.7 Fe	0.05 – 0.1
2496.8 x 2 B	4404.7 Fe	0.001– 0.02
3414.8 Ni	4404.7 Fe	0.005– 0.5
3274.0 x 2 Cu	4404.7 Fe	0.005– 0.2
3412.3 x 2 Co	4404.7 Fe	0.01 – 0.5
3195.0 x 2 Cb	4404.7 Fe	0.005– 0.4
3311.2 x 2 Ta	4404.7 Fe	0.03 – 0.3
3961.5 Al	4404.7 Fe	0.005– 0.3
Silicon and Plain Carbon Steels		
2881.6 x 2 Si	3067.2 x 2 Fe	1.0 – 5.0
2949.2 x 2 Mn	3067.2 x 2 Fe	0.1 – 0.5
2949.2 x 2 Mn	4404.7 Fe	0.01 – 0.5
4254.3 Cr	4404.7 Fe	0.005– 0.5
3414.8 Ni	4404.7 Fe	0.005– 0.5
3170.3 Mo	4404.7 Fe	0.005– 0.5
3961.5 Al	4404.7 Fe	0.005– 0.3
3262.3 Sn	4404.7 Fe	0.005– 0.2
3274.0 x 2 Cu	4404.7 Fe	0.005– 0.2
2496.8 x 2 B	4404.7 Fe	0.001– 0.02

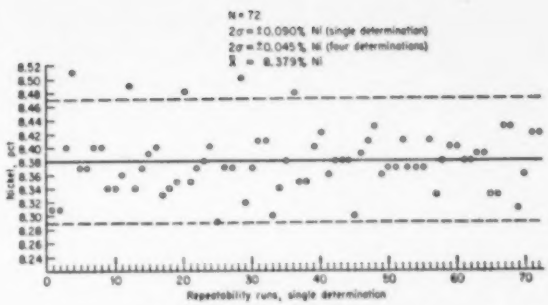
N = 72
2σ = ±0.090% Ni (single determination)
2σ = ±0.045% Ni (four determinations)
X̄ = 8.379% Ni
Nickel, pct
Repeatability runs, single determination

Fig. 1—Reproducibility of nickel in type 304 stainless steel.

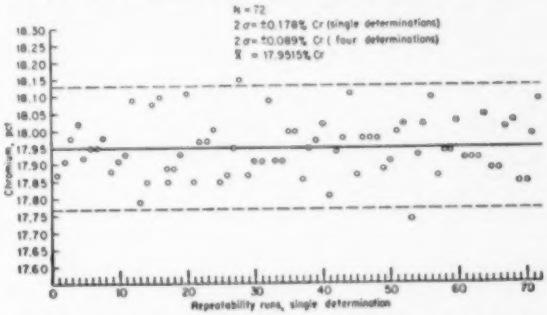
N = 72
2σ = ±0.178% Cr (single determinations)
2σ = ±0.089% Cr (four determinations)
X̄ = 17.9515% Cr
Chromium, pct
Repeatability runs, single determination

Fig. 2—Reproducibility of chromium in type 304 stainless steel.

Reproducibility

To indicate the reproducibility of the quantometric procedure, data were obtained on a standard sample for a period of approximately one month. The sample studied was a type 304 stainless steel. This sample was selected from the group of standard samples used in stainless steel analyses and is therefore representative of the standards used to apply the day to day corrections to the unknowns as outlined earlier in this report. The sample was sparked four times on 18 different occasions for a total of 72 individual sparkings. Figs. 1 and 2 show the reproducibility data obtained for nickel and chromium. Two sigma (2σ) was calculated for each element and found to be ± 0.178 pct for chromium and ± 0.090 pct for nickel. If the average of four determinations are used, which is the number used for daily routine analysis, two sigma (2σ) for each element would be as follows: chromium, ± 0.089 pct; nickel, ± 0.045 pct.

The average concentration (calculated) of the elements in the stainless steel sample used for the reproducibility studies is as follows: chromium, 17.95 pct; nickel, 8.38 pct.

The reproducibility values as determined for any one element would include all the factors which

Table III. Comparison of Quantometric and Chemical Results

No. of Samples	Element	Type of Steel	Concentration Range, Pct	Average Concentration, Pct	Average Deviation from Chemical, Pct
14	Ni	Stainless 410, 430, 430 Ti, 446	0.19 – 0.58	0.33	0.013
70	Ni	Stainless 301, 302, 304, 304L, 305, 17-7 PH	7.20 –11.08	8.94	0.051
133	Cr	Stainless 410, 430, 430 Ti, 446	12.48 –24.95	17.19	0.080
70	Cr	Stainless 301, 302, 304, 304L, 305, 17-7 PH	16.98 –19.76	18.06	0.068
65	Mn	Stainless 410, 430, 430 Ti, 446, 301, 302, 304, 304L, 305, 17-7 PH	0.26 – 1.42	0.56	0.015
95	Si	Stainless 410, 430, 430 Ti, 446, 301, 302, 304, 304L, 305, 17-7 PH	0.30 – 0.97	0.59	0.015
31	Cu	Stainless 410, 430, 430 Ti, 446, 301, 302, 304, 304L, 305, 17-7 PH	0.13 – 0.25	0.17	0.006
39	Ti	Stainless 430	0.080– 0.22	0.14	0.010
29	Si	Silicon	0.95 – 3.47	2.66	0.019
25	Mn	Silicon	0.070– 0.28	0.12	0.004

influenced the quantometric analyses for that element for that run. Some of the factors are operator techniques, instrument errors, errors in interpreting charts, sample preparation, etc.

Accuracy

The accuracy of the quantometric method was determined by comparing the routine results obtained daily on the quantometer with those obtained by wet chemical methods. Comparison of analyses by both the wet chemical and quantometric methods are shown in Table III. These comparisons include all the data obtained on ladle test samples of 203 heats of stainless steel types 410, 430, 430 Ti, 446, 301, 302, 304, 304 L, 305, and 17-7 PH. Also included in the table are results on ladle test samples of 29 silicon steels. The table shows the number of samples from which the comparisons were made, the elements determined in the different types of steel, the concentration range of each element, the average concentration of each element, and the average deviation of the quantometric values from those obtained chemically. The following elements were studied for this paper: nickel, chromium, manganese, silicon, copper, and titanium.

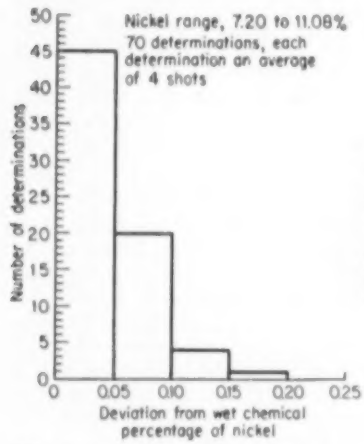

Fig. 3—Quantometric vs chemical values for nickel in stainless steel, ladle test samples. Stainless steel types 301, 302, 304, 304 L, 305, and 17-7 PH.

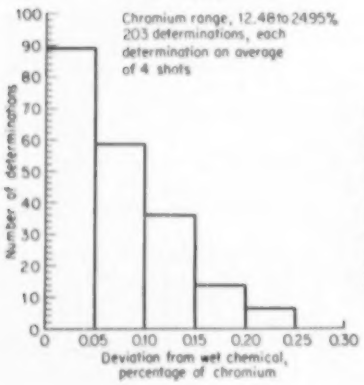

Fig. 4—Quantometric vs chemical values for chromium in stainless steel, ladle test samples. Stainless steel types 410, 430, 430 Ti, 446, 301, 302, 304, 304 L, 305, and 17-7 PH.

Figs. 3 and 4 show a graphical presentation of the accuracy obtained for nickel and chromium in the stainless steel types. For the elements not shown graphically, the following deviations from wet chemical values were obtained:

1—Nickel (types 410, 430, 430 Ti, 446): Only one quantometric value varied by more than ±0.020 pct Ni from the chemical value.

2—Manganese (all types): Six quantometric values deviated by more than ±0.030 pct Mn from the chemical values.

3—Silicon (all types): All quantometric values except one fell within ±0.050 pct Si of the chemical values.

4—Copper (all types): All quantometric values fell within ±0.020 pct Cu of the chemical values.

5—Titanium (type 430): All quantometric values fell within ±0.030 pct Ti of the chemical values.

The data presented in Table III include errors from a number of variables, such as chemical errors, instrument errors, excitation errors, segregation errors. However, the average deviation obtained between the quantometric and chemical determinations show that the accuracy observed on the quantometer is sufficient for the instrument to be used on routine control.

Summary

The use of the ARL production control quantometer has been discussed for the analysis of stainless, silicon, and plain carbon steels. This instrument has afforded a rapid means of analysis for the production control of all types of stainless steel and is also being used in the analyses of final tests from a number of stainless, silicon, and plain carbon steels. Since the installation of the quantometer the laboratory has realized a substantial savings in manpower and time.

The quantometer has been used successfully by the Armco Butler Div. since June 11, 1952. In most cases, the accuracy obtained by quantometric means compare favorably with that obtained by routine wet chemical methods.

Acknowledgments

The author expresses his appreciation to the Armco Steel Corp. for permitting presentation of this paper; to Armco Research Laboratories and to the Butler Chemical Laboratory for their assistance in the analysis of samples.

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[1] M. F. Hasler and B. R. Boyd: *Proc.* Electric Furnace Steel Conference, AIME (1952) **10**, p. 143.

[2] J. F. Woodruff: *Journal* Optical Soc. of Amer. (1950) **40**, p. 192.

Distribution of Manganese Between Slag and Metal Under Reducing Conditions

by J. E. Stukel and J. Cocubinsky

A CONSIDERABLE amount of information is available on the equilibrium distribution of manganese between slag and metal under oxidizing conditions. These data have increased our knowledge of the manganese reactions in the open hearth, ladle, and ingot mold. In contrast, there is a paucity of data on the slag-metal distribution of manganese under reducing conditions. The purpose of the present investigation was to study the equilibrium distribution of manganese between blast-furnace type slags and iron saturated with carbon.

Experimental Method

Furnace: The induction furnace used for the experiments is shown in Fig. 1. A 1 in. pipe, attached to the transite cover, was fitted with a window through which the charge could be observed. Material could be introduced into the furnace through a tee and plug without removing the cover. A graphit cylinder (3 in. ID and 10½ in. long), centered within the induction coil, was used as a heating element. During a test, a zircon plug kept the top of the furnace from becoming too hot from heat radiation. At the bottom, a graphite block on the vertical center line of the furnace and extending 1½ in. into the coil, was used as a support for the graphite crucibles containing the melt. The graphite crucibles, machined from regular carbon electrode stock, had a 2⅝ in. OD with 3/16 in. walls and were 5½ in. long.

The fumes present in the furnace during the early part of a run made optical readings uncertain. A noble metal thermocouple was, therefore, adopted to measure the temperatures. The arrangement used, shown in Fig. 1, allowed the silmanite protection tube to be replaced with a minimum effort.

The thermocouple was calibrated against a standard Pt-Pt-Rh thermocouple in a fused quartz protection tube. The latter was inserted in the molten iron by removing the glass window on the cover. The lower couple was found to be very sensitive to temperature changes. To insure accurate temperature readings, the graphite crucibles were made to fit tightly against the silmanite protection tube. Further, to avoid contamination of the thermocouple by carbon, the silmanite tube was changed and the calibration procedure was repeated at frequent intervals. By using this temperature measuring technique, the furnace was easily held within ±10°C of the desired temperature.

Because of the small free space in the furnace, no attempt was made to control the atmosphere. It

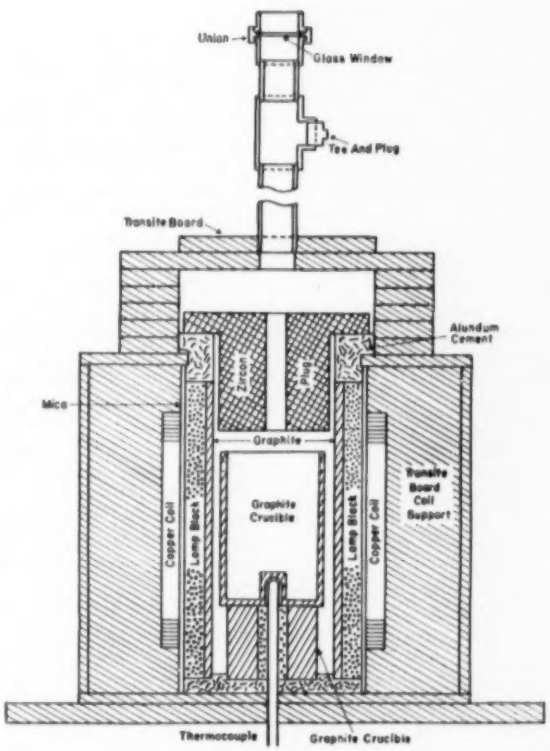

Fig. 1—Schematic drawing of the experimental induction furnace.

J. E. STUKEL, Associate Member AIME, formerly Assistant Professor of Metallurgy and Member of Staff of the Metals Research Laboratory, Carnegie Institute of Technology, is now Development Engineer, Youngstown Sheet and Tube Co., Youngstown, Ohio, and J. COCUBINSKY, formerly Research Assistant, Metals Research Laboratory, Carnegie Institute of Technology, is now Metallurgist, Foster-Wheeler Corp., Carteret, N. J.

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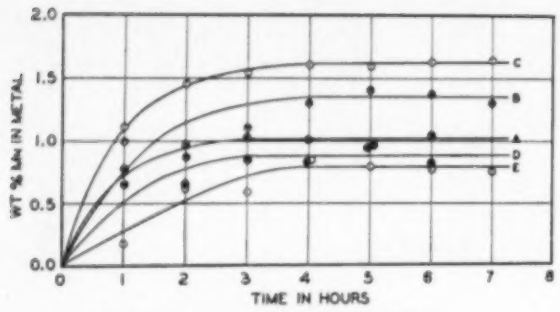

Fig. 2—Time required to reach equilibrium.

was assumed that the CO resulting from the reaction between the hot graphite and the gaseous decomposition products in the charge ($CaCO_3$, H_2SiO_3, MnO_2) would be sufficient to reduce the nitrogen from the entrapped air to relatively low amounts. Further, the CO from the reduction of the silica and MnO would exert a slight positive pressure to minimize air infiltration.

Determination of Equilibrium Time: Hatch and Chipman[1] have shown that a considerable amount of time is required to reach sulphur equilibrium in a system containing carbon-saturated iron and blast-furnace type slags. Their data also show that an even longer time is required for the silicon equilibrium. Because of this, a number of runs were made at random temperatures to determine the time required to reach manganese equilibrium.

The synthetic slags were made by mixing 20 grams of chemically pure MnO_2 with 70 grams of the other slag components; namely, varying amounts of chemically pure quality H_2SiO_3, Al_2O_3, and $CaCO_3$. This mixture was added to a graphite crucible together with 500 grams of ingot iron and a small amount of graphite turnings. The turnings were used to prevent an excessive solution of the crucible walls into the molten iron. The timing was started as soon as the system appeared molten at the desired temperature.

A metal sample was taken every hour during a run by a modification of the Taylor sampler.[7] A ¼ in. pipe was fitted with an aspirator on one end and a threaded couple on the other end. A 4 in. graphite rod, with a ⅛ in. hole drilled through it, was threaded and screwed into the coupling. During sampling, the furnace cover was removed and the sampling rod was inserted through the hole in the zircon plug and into the metal. A solid rod of metal about 3 in. long was usually obtained for chemical analysis. The cover was replaced as soon as the sample was taken.

The results of these tests are plotted in Fig. 2. The scatter in the points, especially during the first 3 hr of a test, might have been minimized if mechanical stirring had been used. Despite this, the plot indicates that equilibrium is reached in about 4 to 5 hr with slags of varying basicities at temperatures at 1300°C and higher. Since the manganese equilibrium was to be determined only from one direction, namely slag to metal, a minimum of 5 hr was considered ample time to reach equilibrium in all succeeding runs.

Determination of the Effect of Slag Composition and Temperature on Manganese Distribution: In order to minimize the effect of segregation, the ingot iron weight was reduced to 250 grams. The manganese dioxide addition was decreased a lesser extent to have a slightly higher manganese content in the metal than in the initial runs. Because the escaping gas mixed the slag phase so well, the weight of slag (minus MnO_2) was increased to 80 grams. As in the previous runs, the slag components, ingot iron, and graphite turnings were all added to the graphite crucible at the start of a test. Although the temperatures were recorded every 15 min, a continual check was made throughout a heat. At the end of 5 hr the crucible was taken out of the furnace and quickly emptied into a cast iron mold. Samples were then prepared for chemical analysis. The results are recorded in Table I.

Experimental Results

Manganese Equilibrium: Using the convention adopted by *Basic Open Hearth Steelmaking* (AIME, 1951 edition), the reduction of manganous oxide from the slag may be represented by the equation

$$MnO + \underline{C} \rightleftharpoons \underline{Mn} + CO. \qquad [1]$$

The equilibrium constant for this reaction may be written

$$K = \frac{A_{Mn}\, P_{CO}}{A_{MnO}\, A_C}. \qquad [2]$$

In a carbon-saturated system the activity of carbon is unity. Despite the possible presence of small amounts of nitrogen in the furnace, the CO pressure, for simplification, is considered equal to one atmosphere. Since the activities of the manganese and manganous oxide are not known, they will be

Table I. Determination of the Effect of Temperature and Slag Composition on the Manganese Distribution under Reducing Conditions

Run No.	Temperature, °C	Final Metal Analysis, Pct		Final Slag Analysis, Pct					
		Mn	Si	CaO	SiO_2	Al_2O_3	MnO	FeO	MgO
1	1448	2.34	0.08	43.44*	39.46	11.77	4.84	0.49	
2	1451	2.25	0.12	43.76*	40.23	10.51	4.73	0.77	
3	1451	2.12	0.10	36.26*	40.91	16.88	5.41	0.54	
4	1450	1.73	0.32	35.37	47.46	9.72	5.52	0.26	
5	1450	1.99	0.12	28.44	38.53	28.72	4.99	0.13	
6	1452	2.06	0.06	33.30	43.72	19.62	4.51	0.22	
7	1453	2.01	0.29	34.95	45.60	14.61	4.75	0.28	
8	1449	1.39	0.06	23.40	53.64	15.21	6.08	0.48	
9	1449	1.60	0.31	28.57	48.38	18.24	5.87	0.57	
10	1449	1.24	0.50	19.59	54.46	18.63	6.40	0.51	
11	1450	1.49	0.57	21.43	49.25	22.27	7.74	0.34	
12	1451	1.24	0.68	24.10	56.26	14.34*	5.11	0.19	
13	1451	1.51	0.61	29.77	53.10	10.24*	6.71	0.18	
14A	1449	0.72	0.17	40.60*	44.81	13.01	1.43	0.15	
14B	1450	1.12	0.14	39.96	45.00	13.00	2.64	0.13	
14C	1449	1.80	0.12	42.80	42.50	11.40	3.64	0.31	
14D	1449	3.95	0.12	38.81	41.00	10.36	9.71	0.16	
15	1450	1.98	0.29	34.76	45.53	8.19	4.85	0.12	4.41
16	1450	2.12	0.31	30.74	45.79	8.32	5.20	0.13	10.73
17	1450	2.09	0.23	25.99	45.36	11.82	4.77	0.10	11.11
18	1449	2.26	0.13	38.10	41.30	11.91	4.24	0.09	5.46
19	1449	2.34	0.13	27.69	41.02	11.71	5.58	0.13	12.72
20	1449	2.35	0.07	34.66	41.11	12.52	4.19	0.13	8.07
21	1449	1.10	0.42	15.59	58.72	18.22	9.12	0.41	
22	1539	2.55	0.74	29.25	36.44	31.29	3.56	0.14	
23	1538	2.56	1.28	42.27	44.26	12.12	3.24	0.19	
24	1542	2.32	0.47	25.82	35.11	26.40	4.53	0.30	
25	1539	2.40	0.21	36.16	37.45	25.14	4.58	0.22	
26	1542	2.45	0.18	31.38	32.23	35.27	3.39	0.18	
27	1541	2.63	0.14	36.73	31.27	31.02	3.12	0.31	
28	1539	2.65	0.18	36.59	35.56	26.34	3.33	0.15	
29	1538	2.50	0.12	39.72	38.26	17.21	4.15	0.14	
30	1538	2.52	0.13	44.90	28.61	12.54	3.82	0.22	
31A	1537	1.88	0.32	38.04	43.00	15.75	3.06	0.18	
31B	1538	1.35	0.42	38.68	43.50	15.85	1.97	0.19	
31C	1538	0.91	0.62	39.10	43.54	16.24	1.17	0.20	
31D	1539	4.40	0.24	37.03	41.34	14.88	6.77	0.20	
32	1538	2.00	1.26	26.27	50.50	17.82	5.18	0.18	
33	1539	1.36	1.06	17.14	55.85	19.80	7.22	0.16	

* Obtained by difference.

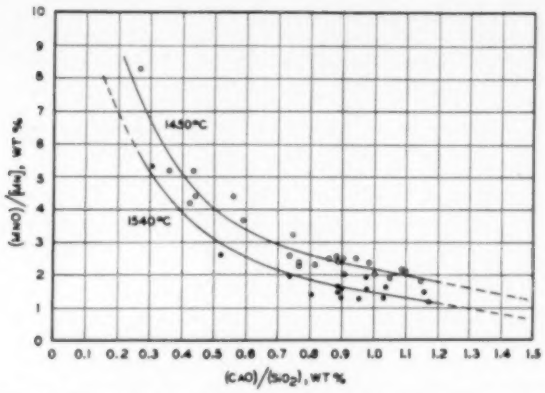

Fig. 3—Effect of the lime-silica ratio and temperature on the manganese distribution.

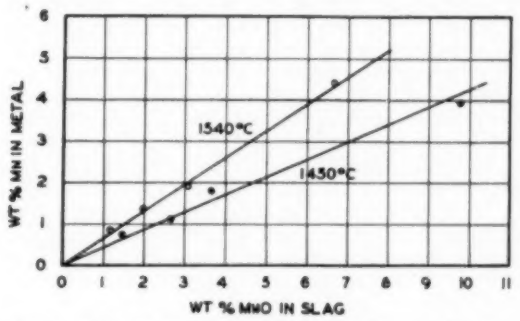

Fig. 4—Effect of the manganese concentration on the distribution ratio.

assumed to be proportional to their respective concentrations. With these conditions in mind, the equilibrium may be written

$$\frac{1}{K} = K' = \frac{(\%\ \mathrm{MnO})}{[\%\ \mathrm{Mn}]}. \qquad [3]$$

A plot of K' as a function of the lime-silica ratio in the slag for the two temperatures used in this study is shown in Fig. 3. The chemically analyzed weight percentages were used to calculate the lime-silica ratios. At a given temperature, an increase in the lime-silica ratio shifts the equilibrium to higher manganese concentrations in the metal. This shift is quite pronounced until the lime-silica ratio approaches 0.8 to 1.0. This change in the manganese distribution ratio is, most likely, due to the higher manganous oxide activity resulting from the neutralization of the silica by the lime. Fig. 3 also shows that at a given lime-silica ratio, a higher temperature decreases the value of K' to favor a greater manganese recovery. Although the alumina in the slags varied from 8.1 to 36.4 pct, it appeared to have no significant effect on the manganese distribution. Thus, the manganese distribution may be described, in the main, by the lime-silica ratio and temperature.

In runs 14A, B, C, and D at 1450°C and in runs 31A, B, C, and D at 1540°C, the manganese addition was varied to determine the effect of concentration on the (MnO)/[Mn] ratio. The lime-silica ratio was relatively constant at each temperature. The results are plotted in Fig. 4. Over the range of manganese concentrations normally found in pig iron, the ratio appears to be independent of concentration.

Runs 15 through 20 at 1450°C were made to determine the effect of magnesia on the manganese distribution. The results are plotted in Fig. 5. In this plot the effect of the slight variation in the (% CaO) + (% MgO)/(% SiO_2) ratio was eliminated by the graphical method of multiple correlation.[8] It appears that magnesia may replace lime up to 13 pct in the slag, on a weight percentage basis, without affecting the manganese distribution.

Silicon Equilibrium: Although the primary subject of this investigation was the manganese distribution, the data also permit a study of the silica reduction. Silicon determinations were made on the metal samples in a number of runs in Fig. 2. It was found that equilibrium was established within 5 hr at 1300°C, but not at 1500°C. Therefore, silicon equilibrium probably was not established in the allotted time in the runs made at 1540°C. There may also be some doubt that equilibrium was approached in the runs made at 1450°C.

Because very few data are available on the reduction of silica from blast furnace slags in the presence of carbon-saturated iron, it was decided to analyze the data to indicate qualitative trends. A plot of the silicon in the metal as a function of the lime-silica ratio in the slag is shown in Fig. 6. This relationship was obtained by eliminating the effect of the alumina in the slag with the aid of graphical multiple correlation. Increasing alumina was found to decrease the silicon in the metal. The manganous oxide in the slag appeared to have no effect on this reaction. In the six runs with magnesia additions, the data did not justify considering the effect of the magnesia separately. For this reason, it was considered equivalent to the lime. The broken line represents the data at 1540°C. As expected, the data at 1540°C showed an even greater scatter than the results at 1450°C.

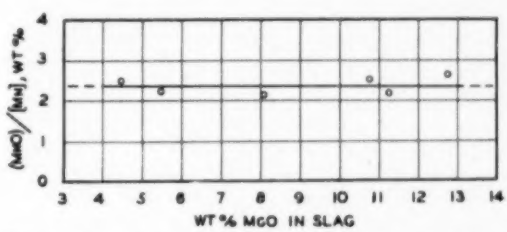

Fig. 5—Effect of magnesia on the manganese distribution.

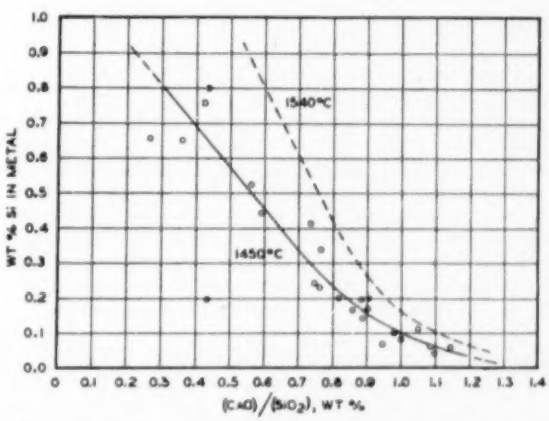

Fig. 6—Qualitative relationship of the lime-silica ratio and temperature on the silicon in the metal.

Iron Content of the Slag: The iron oxide content of the slag was determined after the sample was treated with a magnet to separate any metallic shot. With such low iron contents, the percentage errors in sampling and analysis are high enough to obscure any trends.

Discussion of Results

It has been generally agreed among blast furnace operators that the highest manganese recovery is obtained with a high slag basicity, a high temperature, and a low slag volume. The results in this study provide a more quantitative expression of these observations.

Winkler[4] has reported hot metal temperatures taken at a skimmer in the range of 1425° to 1520°C. The curves in Fig. 3 were extrapolated to include the higher lime-silica ratios (including magnesia) that some blast furnaces use in their operations. The curve for 1540°C predicts the high side of the (MnO)/[Mn] ratios actually obtained on a number of bessemer and basic blast furnaces. In view of the many variables in the iron blast furnace process, this may be considered a reasonably close agreement.

Since the alumina in the slag does not aid the manganese recovery or desulphurization, it would appear feasible to operate the blast furnace with low alumina contents, say 10 pct, and add sufficient magnesia to make an easy working slag.

It is interesting to note that the (MnO)/[Mn] ratio has a decreasing rate of change with basicity in the region of a 1 to 1 CaO/SiO_2 ratio. Similar results have been reported by Crafts and Rassbach[6] for the reducing slags in an electric furnace. It would appear that the dicalcium silicate molecule does not play an important role in reducing slags.

Summary

From the results obtained in this investigation the following conclusions may be reached:

1—The manganese distribution between slag and metal in the blast furnace approaches equilibrium values.

2—The manganese recovery is enhanced by high lime-silica ratios (including magnesia), high temperatures, and low slag volumes. The alumina content of the slag does not appear to affect the manganese distribution.

3—The range of manganese concentrations normally found in pig iron does not affect the (MnO)/[Mn] ratio.

4—Substituting magnesia for lime up to 13 pct on a weight percentage basis does not appear to affect the manganese distribution.

Acknowledgments

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Technical Note

Occurrence of Silicon Carbide in the Fe-C-Si System

by James C. Fulton and John Chipman

BECAUSE of the great importance of the ternary system Fe-C-Si in ferrous metallurgy a considerable amount of attention has been devoted to the phase relations, particularly those occurring in the solid state.[1] The information on graphite solubility

J. C. FULTON, Junior Member AIME, is associated with the Allegheny Ludlum Steel Corp., Watervliet, N. Y., and J. CHIPMAN, Member AIME, is associated with the Dept. of Metallurgy, Massachusetts Institute of Technology, Cambridge, Mass.

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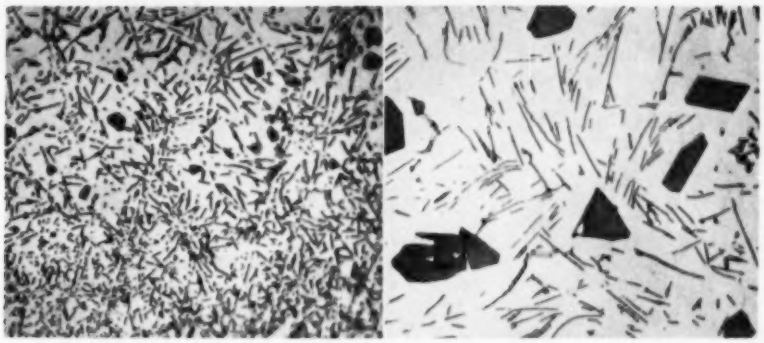

a—Near surface. b—At center.

Fig. 1—Sample containing 19.2 pct Si and 0.70 pct C. Quenched from the melt at 1600°C in Vycor tube. Etched in HF-HNO_3. Larger crystals are β SiC, flakes are graphite, background α Fe. X200. Area reduced approximately 50 pct for reproduction.

in the liquid alloys has been extended recently[2] to temperatures as high as 1690°C and to silicon concentrations up to 23 pct. At concentrations higher than approximately 20 to 23 pct, a second solid phase more stable than graphite with respect to the solution was reported.

This solid phase has now been definitely identified as β SiC. It has been found in residues from the metal and from slags in equilibrium with the metal, and its lattice constants have been found to correspond very closely with published values for β SiC.

Crystals of silicon carbide form rapidly along with graphite during the cooling of such alloys. This is illustrated in the micrographs of Fig. 1, a and b, which were taken from a sample withdrawn in a Vycor tube from the liquid at 1600°C. Both the graphite flakes and the silicon carbide crystals in Fig. 1b, at the center of the sample, have grown considerably larger in size than those in Fig. 1a, at the sample's edge.

Silicon carbide was observed to float out as a crust on high silicon heats made in a graphite crucible. When a slag was present the crust became dispersed in the slag. Samples taken after prolonged holding at constant temperature were analyzed to determine the solubility of silicon carbide in graphite-saturated melts. Average results read from a plot of the data are given in Table I.

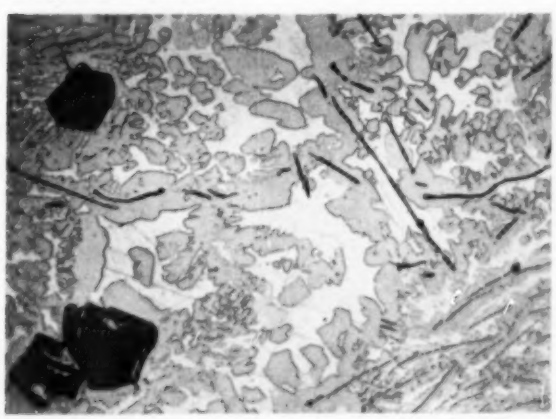

Fig. 2—Sample containing 20.3 pct Si and 0.14 pct C dipped from mushy melt at 1187°C. Etched in HF-HNO_3. X1000. Area reduced approximately 50 pct for reproduction.

Table I. Compositions in Equilibrium with Graphite and β SiC

Temperature, °C	Weight Percent	
	C	Si
1690	0.49	23.7
1600	0.41	23.0
1490	0.33	22.4
1420	0.28	21.9
1290	0.20	20.9
1205	0.15	20.3

A heat which had been held for 2 hr at 1250° and for an additional 2 hr at 1205°C, when cooled slowly in the crucible, exhibited a well-defined arrest at 1187°C. The mass remained in a "mushy" state for 5 min at this temperature and was observed to be completely solid at a few degrees lower temperature. On reheating, the 1187° arrest was repeated and the metal retained its mushy character to about 1210°. A second cooling curve duplicated the 1187° point. A micrograph of a sample dipped from the mushy melt at 1187° is shown in Fig. 2. The large crystals are silicon carbide, the flakes are graphite, the darker gray areas are ϵ FeSi and the lighter areas are α Fe, presumably containing 10 to 11 pct Si.[1]

The exact mode of solidification of this alloy has not been determined. It is complicated by the occurrence of the binary eutectic, Fe-FeSi, at 20 pct Si and 1200°C. Our tentative interpretation is as follows. The 1187° point represents an invariant temperature at which the equilibrium phases are liquid, α Fe, ϵ FeSi, and graphite. At some slightly higher temperature another invariancy must exist in which the equilibrium phases are liquid, graphite, silicon carbide, and either α Fe or ϵ FeSi. This point was not observed in our experiments, probably because of undercooling. Because of the nearness of these two points with respect to both temperature and composition of the liquid, the elucidation of this portion of the ternary phase diagram will require some very careful experimentation.

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Copper Converting Practice at American Smelting and Refining Company Plants

by F. W. Archibald

The American Smelting and Refining Co. has standardized its copper converting practice to attain a maximum unit blister production with a minimum of refractory consumption by careful location of the tuyeres and by applying magnetite coatings on the hard-burned magnesite brick linings.

THE American Smelting and Refining Co. operates four primary copper smelters in the United States with a total of 17 Peirce-Smith type converters; 15 of them are 13 ft in diameter by 30 ft long, and two are smaller. Some details of operations vary with locale; however, fundamentals of design, operation, and maintenance are common to all plants.

All converter shells are 1-in. thick except for one new converter with riding rings on the ends which has a shell thickness of 1½ in. More tuyeres can be installed with rings on the ends and hence more air can be used. Welded construction is replacing riveted. Minor shell repairs are made after each campaign. Principal causes of complete shell replacement are warpage and cracking resulting from localized over-heating. A 13x30 ft shell is being replaced after 34 years of operation and a total production of approximately 750,000 tons of blister. Converters are driven by 80 hp DC motors through company-designed worm gear reducers. Rotation is 0.38 rpm which permits the skimmer to spot the converter quite accurately for skimming slag. At three of the plants, protection against unscheduled air or power failures is afforded by the installation of emergency drives, consisting of auxiliary air motors or storage batteries.

In order to avoid excessive splash out of the converters, the converter mouths are located as far back on the shells as existing flue facilities will permit. At one plant, the back of the mouth is only 13° to the rear of the vertical center line of the converter, whereas it is 28° at another plant. Newly-lined mouth areas vary from 36 to 44 sq ft with effective operating areas about 25 pct less. It is important to keep the converter mouths as clean as possible. Dirty mouths create back pressures in the converters and as a consequence the tuyere air volumes are reduced. Mouths are generally cleaned by bumping with an empty ladle. Small mouth-cleaning rams have been used but unless extreme care is exercised the brickwork may be damaged.

After considerable experimentation, the plants standardized tuyere elevations at 4 to 4½ in. below horizontal center line at the shell with a downward pitch of about 13/16 in. per ft of length. Tuyeres are spaced at 6 in. centers except at the riding rings where there are no tuyeres. With this tuyere location, several of the plants now freeze magnetite slag in the bottoms, fronts, and backs up to the bottom of the tuyeres to control the internal shape of the converter. This has the effect of improving the agitation and mixing so that there is a marked increase in converting speed besides affording protection to the brick lining. Currently, the trend is to increase tuyere diameters from 1½ to 2 in. to increase the air flow. At present, all converters are hand-punched but one converter is being equipped with a set of mechanical tuyere punchers. Rods for punching are ¾ in. hexagonal smelter bar upset to 1⅛ in. and rods for cleaning tuyeres are upset to about 1⅜ in., or larger, depending upon the tuyere diameter. Two of the plants use pneumatic reamers for cleaning the tuyeres between charges to minimize disturbing the coating on the inside of the refractory lining. Most of the puncher's platforms are pneumatically or hydraulically mounted so that a convenient punching position can be maintained regardless of tuyere position.

For normal operations, tuyere air pressures vary from 15 to 13.5 psi, although some cycles such as magnetiting require pressures down to 8 psi. Air requirements vary from an average of 25,000 cfm on the newer installations down to 12,000 cfm on the older ones.

Converter hoods are designed to protect the punchers from sparks, splash or hood accretions as well as to prevent the escape of objectionable

F. W. ARCHIBALD, Associate Member AIME, is Assistant Superintendent, El Paso Smelting Works, American Smelting and Refining Co., El Paso, Texas.

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smoke into the building. The hoods are cleaned periodically to prevent the build-up of large accretions. This is accomplished through cleanout doors in the sides or by using long rams from the converter cranes. At two of the smelters, sulphuric acid is produced from converter gases. This necessitates having very tight fitting hoods and automatic dampers to prevent dilution of the flue gases with air. The dampers are installed in each hood and function whenever the converter turns into or out of the blowing position.

All converters in service are lined with hard-burned magnesite brick at the tuyere line. However, one plant effectively includes a chemically bonded, metal-clad brick as a spacer between tuyere pipes instead of the more conventional tuyere block. At various times, tests have been conducted using other brick in sections of the converters, but, in general, experience has been that best brick life is obtained by magnetite coating hard-burned magnesite brick.

Converter tuyere lines last an average of about 23,000 tons of blister, although one plant is averaging 49,000 tons and has reported a record run of 92,000 tons between repairs. The order of frequency of repair is: first, the tuyere line; second, the mouth; third, the back; fourth, the front; and finally the ends and bottom. Complete relinings occur about every twenty years of operation with one recent relining being made after seventeen campaigns, during which a total of 627,000 tons of blister were produced. Ends, bottoms, and about two-thirds of the backs and fronts are lined with 18 in. brick. The remainder is 15 in. brick except for the mouth section, which is generally 12 in. thick. Mouth sections are built with three or four ring courses across the back and two or three across the front. Currently, converter refractory consumption averages approximately 2.2 lb per ton of blister.

All of the converters are fluxed in the conventional manner through one Garr gun per unit. The Garr gun is usually located on the vertical center line about 4½ ft above the horizontal axis. Use of a small hopper over the Garr gun assists in estimating the weight of flux as charged.

Converter flux analyses vary considerable between smelters because each plant is dependent upon purchased ores for its supply. On the average, the flux analysis is as follows: silica 59 to 72 pct, iron 1.0 to 9 pct, lime 1 to 2 pct, and alumina 1.5 to 10 pct. In some instances, plant flue dusts, converter secondaries, lead plant byproducts, etc., are included with the siliceous materials. Whenever the particle size of the flux can be controlled, a size of +½ in. is desired.

All molten materials are handled in cast steel ladles of 150 cu ft capacity by the overhead cranes. All of the cranes are 60-ton capacity with two auxiliary hoists in addition to the main hoist. Main hoist speeds vary from 28 to 55 ft per min.

To minimize the damage to ladles, two of the smelters have installed swinging ladle bumpers. These consist of 30-ton blocks of cast copper suspended by cables from building girders.

Matte and slag ladles are shelled into large boats which are dumped onto a grizzly. The shells are withdrawn from a hopper under the grizzly into ladles to be used as cold additive in the converters. If the converters cannot utilize all the shells, they are then loaded into railroad cars, crushed, and included with either the converter flux or with the roaster charge.

Copper ladle shells are kept separate and must be used in the converters because unfortunately, a crusher that will efficiently handle pieces of solid copper has not been found.

The normal converter operating cycle begins with an initial charge-up of from 60 to 80 tons of matte. Blowing is commenced as soon as there is enough matte to cover the tuyeres. Flux addition begins immediately after blowing has commenced. Enough flux is added in about 10 min to raise a slag which will have a silica to iron ratio of about 0.54. Cold converter cleanup or plant secondaries are added to this first blow. At one plant, purchased scrap brass and molten matte from a lead dross-reverberatory furnace are treated. Special efforts are made to keep the converter away from a "high flame" during this blow so that removal of impurities can be facilitated by the maintenance of hot, clean slags.

The duration of the first slag blow is approximately 85 min after which 15 to 25 tons of slag are skimmed off. The skimmer rabble-tests the slag for the presence of matte or white metal. This slag may analyze as follows: copper 2.3 pct, silica 26.2 pct, total iron 48 pct, lime 1.7 pct, alumina 4.5 pct. The magnetite content will vary from 15 to 22 pct.

Succeeding slag blows are similar except that the quantities of matte added are progressively reduced so that the converter is not over-filled to the point where it will splash. The number of slag blows as well as the duration of each blow varies with the matte grade, but with a 40 to 45 pct copper matte four separate slag blows are made and a total of 160 tons of liquid matte are added for a 60 to 70 ton charge of blister.

Copper oxide slag is added whenever convenient. If molten, such slag must be added very slowly because of the violence of the reaction of copper oxide with matte or white metal. At some plants, where the sliding hood sections will allow clearance, this slag is added to the converter while it is blowing which greatly reduces the hazards of an explosion or blow-out.

There is usually no cold cleanup added to the last blow before going on finish, except for some clean scrap copper, in order to ensure hot, fluid slag at the completion of the blow.

During the slag blows, the converter operating temperatures are maintained at approximately 2250°F, although the temperatures immediately after the flux additions drop to 2100°F, or less. The slag temperatures are approximately 2150°F. It is returned to the reverberatory furnaces except the slag from the final slag blow, which is relatively high in copper and is returned, when possible, to another converter.

Throughout the slag blows, the silica to iron ratio of 0.54 is maintained but the magnetite content of the slags tends to increase progressively. In most cases, all punching is in response to indicating flow meters at each puncher's platform. Air consumption is about 60,000 cu ft per ton of matte converted. Tuyeres are immersed from 14 to 18 in. in the bath during slag blows.

Standard finishing practice is for each converter to process its own white metal rather than to combine charges from several converters into one. The plants have found that a finish of from 60 to 75 tons of blister results in best efficiency and minimizes

damage to brickwork. It has been noted that with larger charges, over-heating occurs.

The finish blow commences after the white metal has been skimmed clean of slag or any unsmelted silica that may remain on the bath. The normal charge consists of 85 to 110 tons of white metal with a copper content of approximately 74 pct. The tuyeres are immersed from 12 to 15 in. After starting the finish blow, the skimmer carefully observes the flame and makes occasional tests with a rod through the tuyeres to look for the presence of copper. Operating temperatures of 2300°F are slightly higher than on slag blows. They are controlled by the addition of clean scrap copper from time to time. About half as much punching is required during the finish blow as compared to the slag blows.

Because of the presence of small amounts of undesirable lead in the blister copper, it is standard practice to overblow the charge so that a portion of the copper is oxidized. The overblow stage normally begins about 3 hr after the finish blow started or when a "flat" blister has been reached. At the beginning of the overblow, approximately one ton of lead-free siliceous flux is added through the Garr gun. This silica forms a slag with the oxidized copper and minimizes the corrosive action of the copper oxide on the magnesite brick or coating. The overblow lasts 20 to 30 min and from 5 to 7 tons of copper are oxidized. Total air consumption averages 150,000 cu ft per ton of blister produced.

After the overblow period, the oxidized slag may be skimmed at the converter or may be held in the converter with a silica bank. All blister is transferred by the cranes from the converters directly to the casting furnaces except at one plant where an intermediate holding vessel is used. Additional oxide slag is generally skimmed off the blister at the casting or holding furnaces. Copper in the blister will approximate 98.4 pct.

It is standard practice at all company smelters to magnetite for the protection of the interior surfaces of the converter refractory lining. A converter in normal operation is coated wherever required; a newly repaired converter, however, is not coated until it has finished three or four charges of blister or until the brick surfaces are roughened up and the brick joints are sufficiently pronounced so that the magnetite will adhere.

After a converter has finished a charge of copper and visual inspection through the mouth reveals exposed brick or brick joints, a magnetite coating is started. The converter must be clean of any siliceous material because silica interferes with formation of magnetite and frequently results in dangerous foaming. If any is present, a "wash out" blow must be made. The Garr gun is plugged with clay to prevent fouling. From 50 to 60 pct of the matte normally required for a first slag blow is charged for magnetite coating and blowing commences without addition of any other materials. The tuyeres are submerged from 4 to 6 in. and with a 40 pct copper matte, magnetite begins to form after 1 to 1½ hr of blowing. The presence of magnetite is indicated by large quantities of brilliant iron sparks showing in the flame. Converter temperatures will rise sharply and may approach 2800° to 3000°F. Care must be exercised that dangerous over-heating with resultant damage to brickwork does not occur. Clean, cold matte can be added at this point to cool the converter if necessary. Once the magnetite starts to form, continuous punching is necessary or the tuyeres will rapidly block off. Also, the skimmer must observe the progress of the coating frequently by turning the converter out of the stack. If large knobs or "shankers" are forming on the tuyeres, the converter is blowing too lightly and the tuyeres must be submerged deeper into the bath. Conversely, if on observation, through the mouth, it is noted that no coating is adhering onto the tuyere line, the tuyeres have been submerged too far and must be brought out slightly. After blowing from 2 to 2½ hr or when blister copper appears on the test rod the converter is turned out, and the tuyeres are cleaned. If a thicker coating is desired, another 10 to 12 tons of matte are added and the process repeated. In most cases, the thickness of coating will be from 1½ to 2 in. After the supplementary blow is completed, the tuyeres are cleaned again. At this stage, the slag is practically pure magnetite and may be in a semifused or "granulated" condition. This is the slag which may be used for freezing in other converters as mentioned previously. The normal slag blow then commences with the addition of two or three ladles of matte (24 to 36 tons) and sufficient flux to make satisfactory slag. Converter temperatures will remain above normal for several hours and the resulting slags will be about twice as high in magnetite as for normal blows.

The normal converter cycle from original charge-up to finish is delayed only about 2 hr by magnetiting because the original matte has been advanced to white metal and higher converter temperatures exist.

In order to maintain operations at peak levels, it has been necessary for the smelters to institute programs of preventive maintenance. All departmental equipment is inspected daily for mechanical defects with particular emphasis placed on crane inspection. To meet production requirements, brickwork repairs to a converter are often postponed. Temporary repairs using basic cement grouting are effective and are performed with only a few hours delay.

Every effort is made to provide good working conditions. Fresh, cooled air from desert-type coolers is supplied to the punchers and skimmers wherever sufficient natural ventilation is lacking. Matte ladle hoods are installed at all reverberatory tapping positions to collect and vent obnoxious gases to atmosphere outside of the working areas. At the company's largest operation where the converter aisle is relatively crowded, the crane cabs are completely air conditioned. This is accomplished by using bag filters and activated carbon for purification plus Freon compressors for refrigeration.

In keeping with company practice, very active safety programs are constantly maintained. As a safety precaution, all bails and crane hooks are stress-relieved by annealing at six months intervals. Safety glasses with the necessary optical corrections are provided to employees. They may purchase safety shoes at cost. Some of the plants have found that whitewashing the ladles, platforms, walls, etc., contributes to safer operations besides greatly enhancing the appearance of the departments. Good housekeeping practices are stressed and in the last few years mechanical front-loaders have been used very effectively for keeping some of the working areas clean.

A Quantitative Measure of Temper Embrittlement

by Norman Brown

From the theories of flow and fracture it is shown that the difference in reciprocals of the transition temperatures (°K) is a quantitative measure of temper embrittlement. Experimental data are given which support this conclusion.

STUDIES of temper embrittlement have been made with various viewpoints; some involved a study of the kinetics of the reaction,[1, 2, 3] others were concerned with changes in the mechanical properties,[4, 5] and still others investigated the structural differences between the embrittled and unembrittled states by means of the microscope,[6] X-ray,[7] measurements of electrode potential,[8] and electrical resistivity.[9] In all these studies, the difference in impact properties was the criterion for distinguishing between the embrittled and unembrittled states. By common agreement the difference in transition temperatures is now taken as a measure of the degree of embrittlement, and this measure is used as the quantitative measure upon which studies of the phenomenon are based.

Vidal and Jolivet[10] suggested that the degree of embrittlement was the same whether measured by impact or by a slow bend test. Their conclusion was based on tests at constant temperature. Jaffe and Buffum[11] using the impact and slow bend test showed that, for a given degree of embrittlement, the difference in transition temperatures varied with the method of testing. Hultgren and Chang[12] showed that the difference in transition temperature produced by the V-notched Charpy impact test was not the same as the value produced by the keyhole-notched impact test. This is a very fundamental point because there can be no true measure of temper embrittlement unless it is invariant with respect to the test method.

Theory

The purpose of this analysis is to show how the transition temperature depends on the stress distribution and the state of the material. The criterion for fracture may be stated as follws:

$$f_F(\sigma_1, \sigma_2, \sigma_3) = F \qquad [1]$$

N. BROWN is Assistant Professor, Dept. of Metallurgy, University of Pennsylvania, Philadelphia.

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where f_F is some function of the principle stresses and F is a critical condition depending on the state of the metal. Similarly yielding occurs when

$$f_Y(\sigma_1, \sigma_2, \sigma_3) = Y \qquad [2]$$

where $f_Y(\sigma_1, \sigma_2, \sigma_3)$ is another function of the principle stresses and Y is a critical value depending on the state of the material. There is some controversy as to the exact form of the function $f_Y(\sigma_1, \sigma_2, \sigma_3)$ but most experimental data give the following criterion:[13]

$$f_Y(\sigma_1, \sigma_2, \sigma_3) = (\sigma_1 + \delta_2\sigma_2 + \delta_3\sigma_3) \qquad [3]$$

where δ_2 and $\delta_3 = \pm 1$ or 0.

Combinations of δ_2 and δ_3 give the various criteria such as the normal stress, the hydrostatic tension, and the maximum shear stress laws.

Agreement is general that the octahedral shear stress law adequately describes the yield condition where

$$f_Y(\sigma_1, \sigma_2, \sigma_3) = [(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + \sigma_3 - \sigma_1)^2]^{1/2}. \qquad [4]$$

If $\sigma_1 > \sigma_2 > \sigma_3$ and $\sigma_2 = K_2\,\sigma_1$ and $\sigma_3 = K_3\,\sigma_1$ then for fracture

$$\sigma_1\,[1 + \delta_2 K_2 + \delta_3 K_3] = F \qquad [5]$$

and for flow

$$\sigma_1\,[(1 - K_2)^2 + (K_2 - K_3)^2 + (K_3 - 1)^2]^{1/2} = Y \qquad [6]$$

where the bracketed factor will be called the design factor and depends on the stress distribution. Whether the metal decides to yield or fracture is given by the condition that

$$\frac{F}{1 + \delta_2 K_2 + \delta_3 K_3} = \frac{Y}{[(1 - K_2)^2 + (K_2 - K_3)^2 + (K_3 - 1)^2]^{1/2}}. \qquad [7]$$

The effect of temperature and strain rate upon Y has been studied by various investigators. It has been shown by MacGregor and Fisher[14] that

$$Tm = T(1 + K \ln \dot{\epsilon}/\dot{\epsilon}_o) \qquad [8]$$

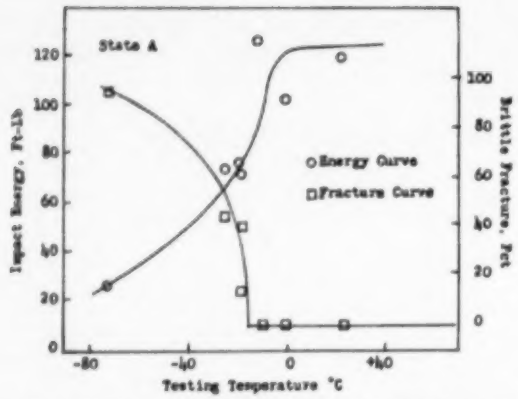

Fig. 1—Typical transition curves. Standard V-notched Charpy tests.

where $$Y = f(\epsilon, Tm). \quad [9]$$

Zener and Hollomon[15] have proposed the parameter

$$Z = \epsilon\, e^{q/T} \quad [10]$$

$$Y = f(\epsilon, Z) \quad [11]$$

where ϵ is the strain, T is the temperature, and K, ϵ_0, and q are constants of the material.

The parameter by MacGregor has been applied to a wider range of temperatures than the one proposed by Zener and Hollomon. The simpler parameter by Zener will be used because it satisfactorily describes the behavior of steel at low temperatures.

The function, $f(\epsilon, Z)$ may be obtained from the following observations on steel:

$$Y = A'\,\epsilon^{n} \quad (T = \text{constant}) \quad [12]$$
$$(\dot{\epsilon} = \text{constant})$$

and $$Y = B'\,\dot{\epsilon}^{n'} \quad (T = \text{constant}) \quad [13]$$
$$(\dot{\epsilon} = \text{constant}).$$

A', n, B', and n' are material constants.

By transforming Eqs. 10, 11, 12, and 13 to functions of log Y and taking the most general linear combination of these equations the following is obtained:

$$\ln Y = A + B \ln\epsilon + C\,(\ln\epsilon + q/T) + D \ln\dot{\epsilon}\,(\ln\dot{\epsilon} + q/T) \quad [14]$$

where A, B, C, D and q are constants of the material. Other mechanical equations of state have been proposed but they offer no advantages over Eq. 14 when applied to steel in the low temperature range. This derivation of a mechanical equation of state has been presented by Dorn.*

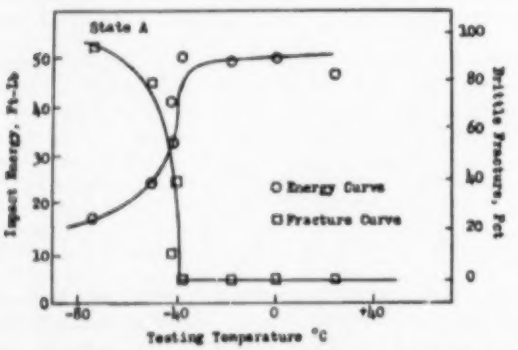

Fig. 2—Typical transition curves. Thin V-Notched Charpy tests.

* The derivation follows the lecture notes of Professor J. E. Dorn at the University of California.

This analysis will be applied to the transition temperature for ductile to brittle fracture as exhibited by temper embrittled steels. A given degree of embrittlement is produced by heating a tempered martensite for various lengths of time at a lower temperature (about 500°C) than for the original tempering (650°C). It is assumed that the embrittling treatment does not affect the stress-strain curve. It is not unusual for a steel to have different fracture properties and yet exhibit the same stress-strain curve. This means that the same material constants in Eq. 14 apply both to the embrittled and unembrittled states.

The critical condition for fracture is determined by varying the testing temperature and keeping the design factor constant. This critical condition is taken as the first appearance of brittle fracture. The temperature of testing corresponding to this critical condition is called the transition temperature. By combining Eqs. 7 and 14 the following equations hold for the embrittled and unembrittled states:

$$\ln F_E = A + B \ln\epsilon_E + C\,(\ln\dot{\epsilon}_E + q/T_E) + D \ln\epsilon_E\,(\ln\dot{\epsilon}_E + q/T_E) + \ln \frac{[1 + \delta_2 K_2 + \delta_3 K_3]}{[(1-K_2)^2 + (K_2 - K_3)^2 + (K_3 - 1)^2]^{1/2}} \quad [15]$$

$$\ln F_U = A + B \ln\epsilon_U + C\,(\ln\dot{\epsilon}_U + q/T_U) + D \ln\epsilon_U\,(\ln\dot{\epsilon}_U + q/T_U) + \ln \frac{[1 + \delta_2 K_2 + \delta_3 K_3]}{[(1-K_2)^2 + (K_2 - K_3)^2 + (K_3 - 1)^2]^{1/2}}. \quad [16]$$

The subscripts U and E refer to the unembrittled and embrittled states respectively. For specimens of the same geometry and fractured by the same test method, Eqs. 15 and 16 are combined with the result that

$$q\left[C\left(\frac{1}{T_U} - \frac{1}{T_E}\right) + D\left(\frac{\ln\epsilon_U}{T_U} - \frac{\ln\epsilon_E}{T_E}\right)\right] = \ln\frac{F_U}{F_E} - B\ln\frac{\epsilon_U}{\epsilon_E} - C\ln\frac{\dot{\epsilon}_U}{\dot{\epsilon}_E} - D\ln\left(\frac{\epsilon_U^{\ln\dot{\epsilon}_U}}{\epsilon_E^{\ln\dot{\epsilon}_E}}\right). \quad [17]$$

Since the same design factor and the same method of loading was used in obtaining Eq. 17, $\dot{\epsilon}_U/\dot{\epsilon}_E = 1$. Observations of specimens fractured at the transition temperature do not indicate any difference in strain prior to fracture between the embrittled and unembrittled states. Because the strain prior to fracture is so nonhomogeneous, these observations are limited to a visual inspection of the fractured specimen. For a given design factor there was no indication that the overall strain at a given amount of brittle fracture is any different in the embrittled and unembrittled states. More significantly the surface at the point of brittle fracture appeared to be the same for a given design factor irrespective of the transition temperature. There is no reason to assume that the ratio of ϵ_U/ϵ_E in Eq. 17 is substantially different from unity.

It is to be noted that $C + D \ln\epsilon = n'$. An evaluation of the constants C and D from true stress-true strain data[14] in the low temperature range shows that D is very small compared to C. Within the limits of measurement D is practically zero and C is about 0.01. It is also to be pointed out that the maximum

Table I. Transition Temperatures °K

Investigators	Test	State A	State B	State C	State D
Present	Std V-notched impact	256	332	403	453
	Rectangular section, V-notched impact	233	306	375	406
	Slow bend, V-notched	229	303	343	409
	Rectangular section, U-notched impact	197	245	282	
Jaffe and Buffum[11]	Std V-notched impact	188*	258**	263†	
	Slow bend, V-notched	167	223	233	
Hultgren and Chang[12]	Std V-notched impact	233††	421‡		
	Keyhole impact	193	318		

* State A — 3140 steel, tempered 675°C for 1 hr, water quenched.
** State B — 3140 steel, tempered 675°C for 1 hr, water quenched, re-tempered at 480°C for 48 hr.
† State C — 3140 steel, tempered 675°C, slow cooled.
†† State A — 3312 steel, tempered 650°C for 1 hr, water quenched.
‡ State B — 3312 steel, tempered 650°C for 1 hr, slow cooled.

natural strain at fracture in the low temperature range generally does not exceed 1.3 for commercial ferritic steel and does not exceed 2.0 even for the highest purity ferrites. Thus the term,

$$D\left(\frac{\ln\epsilon_U}{T_U} - \frac{\ln\epsilon_E}{T_E}\right),$$

may be eliminated from Eq. 17 without affecting the remainder. Thus, Eq. 17 reduces to

$$\frac{1}{T_U} - \frac{1}{T_E} = \frac{\ln\dfrac{F_U}{F_E}}{qC} \qquad [18]$$

The fracture stress is known to change with both temperature and strain prior to fracture. The effect of temperature is usually considered to be small compared to its effect on flow. It appears that the fracture stress of single crystals[16] is independent of temperature. To a first approximation it may be assumed that the ratio F_U/F_E is independent of temperature. This assumption has the effect of over estimating this ratio. The effect of strain on fracture stress is not well established but it appears that for an isotropic material like tempered martensite the fracture stress increases slowly as the strain prior to fracture increases. This will have no effect on the F_C/F_E ratio for a given test condition but it may slightly change the F_U/F_E ratio when the type of test is changed. However for first order approximation it is assumed that F_U/F_E is independent of the test conditions. Since little is known about the effect of strain rate on the fracture stress, it is also assumed that F_U/F_E is independent of strain rate. This means that the difference in the reciprocals of the transition temperatures in degrees Kelvin only depends on material constants. Such a measure of the degree of temper embrittlement is an invariant with respect to the test method.

The following experimental work was done to test whether $(1/T_U) - (1/T_E)$ is invariant as predicted above.

Experimental Work

The steel used was commercial SAE 3312 received in the form of hot rolled ⅞ in. square rods and later reduced to a 9/16 in. square cross section by hot forging. The steel had the following composition: C, 0.13 pct; Mn, 0.5; Ni, 4.2; Cr, 1.3; Si, 0.25; P, 0.012; S, 0.026; Mo, 0.02; and Al, 0.02.

The steel was austenitized at 910°C for 1 hr and then water quenched. The as-quenched structure was 100 pct martensite throughout the 9/16 in. rods and the hardness was 39 to 41 Rockwell C. Tempering was done at 650° ±3°C for 2 hr. All specimens were divided into four groups in order to produce various degrees of embrittlement. The subsequent reheating was done in a salt bath, and the various states of embrittlement were produced by the following treatments: state A received no further treatment; state B, 2/3 hr at 525°C and water quenched; state C, 3 hr at 525°C and water quenched; and state D, 47 hr at 525°C and water quenched.

The hardness of the steel, measured on a Rockwell C scale, in each state was as follows: A, 21.5; B, 20.5; C, 21.0; and D, 21.0.

For each state the transition temperature was found by the following test methods: Test 1 was a standard V-notched Charpy impact test using a 240 ft-lb Sonntag machine with striking velocity of 16.8 ft per sec. Test 2 was the same as Test 1 except the specimen had a rectangular cross section where the dimension parallel to the notch was one-half the standard dimension; all other dimensions were the same as for the standard V-notched Charpy specimen. Test 3 was a slow bend test, bending the standard V-notched Charpy specimen as a simply supported beam under a central load. The rate of deflection was 0.012 in. per min. Test 4 specimen was 0.185x0.394x2.165 in.; the notch was a circular groove of ⅜ in. diameter and 0.100 in. deep. The groove was parallel to the small dimension. The specimen was fractured as in Test 1.

Typical curves presenting the experimental data are shown in Figs. 1 and 2. The percentage of ductile fracture was estimated by comparison with photographs furnished by Watertown Arsenal. The transition temperature was taken as the lowest temperature at which 100 pct ductile fracture was obtained. The reason for a fracture rather than an energy criterion was twofold: 1—a fracture criterion had greater fundamental significance as pointed out by Orowan,[15] and 2—the 100 pct ductile criterion was a rather sharply defined point and it is not sensitive to slight differences in notch geometry which might be introduced by machining.

Table II. Analysis of Data

Investigators	Test	$T_B - T_A$	$\frac{1}{T_A} - \frac{1}{T_B}$	$T_C - T_A$	$\frac{1}{T_A} - \frac{1}{T_C}$	$T_D - T_A$	$\frac{1}{T_A} - \frac{1}{T_D}$
Present	Std V notched	76	0.00090	147	0.00142	197	0.00170
	Thin V-notched	73	0.00102	142	0.00162	173	0.00183
	Slow bend	74	0.00107	114	0.00145	180	0.00192
	Thin U-notched	48	0.00100	85	0.00153		
Jaffe and Buffum[11]	Std V-notched	70	0.00144	75	0.00152		
	Slow bend	56	0.00150	66	0.00170		
Hultgren and Chang[12]	Std V-nouched	188	0.00191				
	Keyhole notched	125	0.00204				

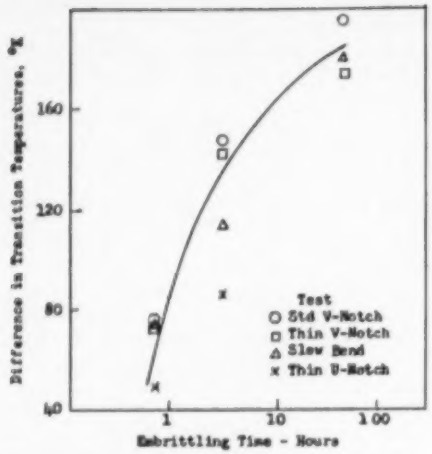

Fig. 3—Degree of embrittlement vs embrittling time at 525°C for SAE 3312 steel.

The transition temperatures are presented in Table I along with all the pertinent transition temperatures available in the literature.

Analysis of Data

It was suggested by the theory that the difference in the reciprocals of the transition temperatures for two given states should be independent of the test method. Since the existing measure of the degree of temper embrittlement is simply the difference in transition temperatures, it would be well to see how the existing measure compares with the proposed measure. The calculations are given in Table II.

The percentage of experimental error shown in Table III was based on a standard deviation of ±2°C in measurement of the transition temperature. This error is based on the observation that about 70 pct of the experimental observations are favorable in a range of ±2°C. Thus, 70 pct of all tests within 2° above the transition temperature showed no brittle fracture, and within 2° below the transition temperature about 70 pct of the fractures were partly brittle. This estimate of the uncertainty in the transition temperature is about the same as the uncertainty in the data presented by other investigators. As an example, the standard deviation of the difference in the reciprocals of the transition temperatures of states A and B of the present investigation was based on the mean of four tests. The standard deviations in the data from the other investigators were based on only two tests. Finally the mean value of the errors in all investigations was determined. The results show that the difference in reciprocals is invariant within the limits of experimental error and that the simple difference deviates significantly from being invariant with respect to the test method.

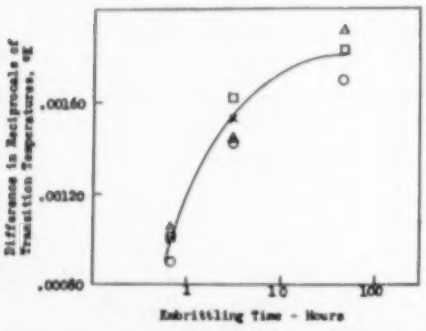

Fig. 4—Degree of embrittlement vs embrittling time at 525°C for SAE 3312 steel.

Discussion

It has been shown experimentally for temper martensite that a given degree of temper embrittlement gives a difference in the reciprocals of the transition temperatures which is independent of the test method. As shown by the theory this invariance will not hold for steels whose stress-strain curves are different or whose fracture stress depends on prior strain in a significantly different manner.

The proposed measure for temper embrittlement may be used to predict transition temperatures. The degree of embrittlement is conveniently measured by the standard Charpy impact test. Then the transition temperature for one state need be determined with a different design factor; the transition temperature of the other state may be calculated for this same design. This may be very useful if the design is complicated, for example part of a ship or a gun mount.

Table III. Error Analysis

Investigation	States	Difference in Reciprocals	Percent Standard Deviation from Mean: Simple Difference	Percent Standard Deviation from Mean: Experimental
Present	A and B	6	17	6
	A and C	4	20	3
	A and D	5	6	2
Jaffe and Buffum[11]	A and B	2	11	6
	A and C	6	6	6
Hultgren and Chang[12]	A and B	3	20	3
Average error for all investigations, pct		4	13	4

In studying the kinetics of the embrittling process it is useful to find out how the degree of embrittlement varies with time during isothermal embrittlement. When the difference in transition temperatures is plotted as a function of time, a different curve is obtained for each type of test used, as shown in Fig. 3. This introduces an ambiguity in the study of the embrittling process. However, if the difference in reciprocal of the transition temperature is plotted against embrittling time, as shown in Fig. 4, a curve is obtained which is essentially independent of the test method and better reflects the nature of the reaction. The difference between the two curves does not result from a difference in scales because the same percentage error was involved in each case.

The difference in the reciprocals of the transition temperatures gives a measure of the ratio of the fracture stresses of the unembrittled and embrittled states. From Eq. 18

$$\ln \frac{F_v}{F_z} = qC \left(\frac{1}{T_v} - \frac{1}{T_z} \right).$$

A value of 0.01 for C is obtained from the work of MacGregor and Fisher.[14] For steel, 5000 is an average for q.[17] The observed values of $(1/T_v) - (1/T_z)$ range from about 0.001 to 0.002. This gives values of F_v/F_z ranging from 1.1 to 1.25. This is a very

reasonable result.[13] In the case of tempered martensite direct measurements of the stress for brittle fracture have yet to be made because the transition temperature is too low for simple tension. In the absence of such data the above method is the only one available for quantitatively determining how much temper embrittlement decreases the fracture stress.

Conclusions

1—The essential difference between the embrittled and unembrittled state lies in the stress required to produce brittle fracture.

2—The difference in the reciprocals of the transition temperatures is an invariant with respect to the test method.

3—The simple difference in transition temperatures is not an invariant measure of the difference between the embrittled and unembrittled states.

Acknowledgment

The author thanks Professor E. R. Parker under whose guidance the research was conducted. Professor J. E. Dorn provided many stimulating discussions.

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Technical Note

On the Effects of Oxygen on Molybdenum

by R. E. Maringer and A. D. Schwope

IT has been recognized that oxygen in minute quantities is extremely detrimental to the room-temperature ductility of molybdenum. Early fractographic studies[1] of the cast metal indicated that oxides are formed preferentially at the grain boundaries. Additional verification of this preferential segregation has been obtained from internal friction studies on polycrystalline molybdenum (average grain diameter varied from 0.039 to 0.065 mm) and from observations of the effect of oxygen on specimens of extremely large grain size.

R. E. MARINGER and A. D. SCHWOPE, Associate Member AIME, are associated with Battelle Memorial Institute, Columbus, Ohio.

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Specimens A, B, and C, of 0.040 in. diameter commercial molybdenum wire (Lot FW 13-40) were heat treated in the vacuum furnace described by Few and Manning[2] according to the specifications given in Table I. Internal friction studies of these specimens were carried out using a low frequency (approximately 1 cycle per second) torsional pendulum completely enclosed in a vacuum tank. As the pressure of oxygen in the heat treating atmosphere changed, the portion of the internal friction vs temperature curves ascribed to grain boundary relaxation changed correspondingly, Fig. 1. The decrease in the damping capacity at high temperatures as the pressure of oxygen in the heat treating atmos-

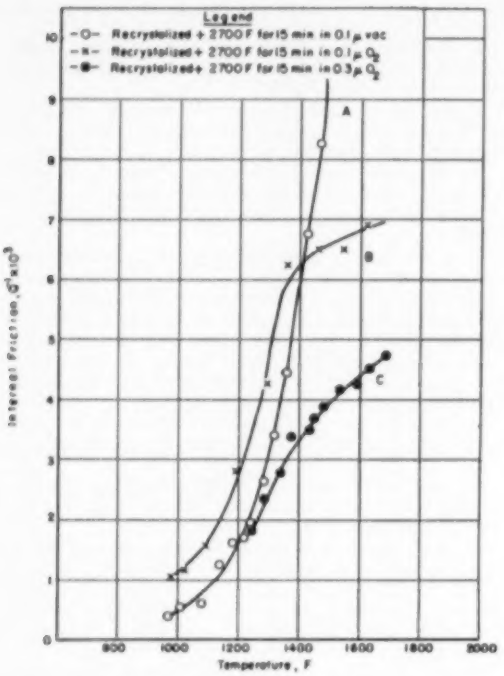

Fig. 1—Variation of internal friction vs temperature curves with heat treating atmospheres.

phere increases indicates the formation of an increasing amount of oxide at the grain boundaries.

The severe grain boundary embrittlement which accompanies this precipitate was demonstrated through the use of large grain size specimens. Specimens D and E from Lot FW 25-40, which exhibited exaggerated grain growth at high temperatures, were heat treated according to the specifications given in Table I. Specimen D, subjected to recrystallization and solid-state purification (heat treatment at high temperature in vacuum tends to remove most of the oxygen present) only, was quite ductile, having 22.8 pct elongation in tension and pulling down to a knife edge. Specimen E, however, which was impregnated with oxygen after recrystallization, was too brittle to be tested in tension. It fractured at a grain boundary during mounting, Fig. 2. The individual grains remained ductile and could be bent around a sharp radius. The strain in the outer fibers of the specimen shown in Fig. 3 is about 40 pct.

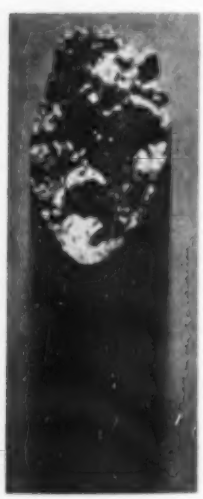

Fig. 2—Intercrystalline fracture of oxygen-impregnated molybdenum. X20.

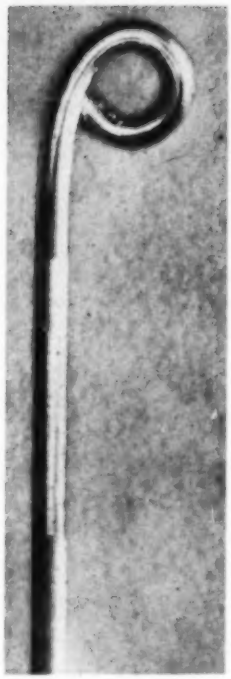

Fig. 3—Ductility of single crystal of oxygen-impregnated molybdenum. X5.

Table I. Heat Treatment of Molybdenum Specimens

Specimen	Lot No.	Recrystallization and Solid-State Purification: Temperature, °F	Time, Min	Atmos.	Oxygen Impregnation: Temperature, °F	Time, Min	Atmos.
A	FW 13-40	3800	15	0.1 μvac.	2700	15	0.1μvac.
B	FW 13-40	3800	15	0.12μvac.	2700	15	0.1μO_2
C	FW 13-40	3800	15	0.1 μvac.	2700	15	0.3μO_2
D	FW 25-40	3800	15	0.02μvac.	—	—	—
E	FW 25-40	3800	15	0.01μvac.	2700	15	0.2μO_2

The embrittling effect of small pressures of oxygen during heat treatment suggests that the so-called "recrystallization embrittlement" of molybdenum is actually oxygen embrittlement. A worked structure or a small grained structure tends to reduce the embrittling effect of the oxygen present by dispersing the oxides over a larger surface area. Thus a large grained cast ingot may be embrittled by a small amount of oxygen while a worked or small grain size material containing the same amount of oxygen may exhibit appreciable ductility.

Acknowledgment

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Constitution and Mechanical Properties Of Titanium-Hydrogen Alloys

by G. A. Lenning, C. M. Craighead, and R. I. Jaffee

Hydrogen forms a beta-stabilized system with titanium, with a beta eutectoid at about 300°C and 44 atomic pct H_2. The solid solubility of hydrogen in alpha decreases from about 8 to about 0.1 atomic pct from 300°C to room temperature. Hydrogen has little effect on tensile properties, but decreases notch-bar toughness to a large degree. This latter effect appears to be the result of increased notch sensitivity.

PRIOR to this investigation, only a small amount of information was available on the effect of hydrogen on the mechanical properties of titanium. The effects of the other interstitially soluble elements, carbon, oxygen, and nitrogen, had received detailed study. Jaffee and Campbell[1] indicated that hydrogen up to 1 atomic pct did not affect tensile properties deleteriously. Pitler[2] indicated that 1 atomic pct H_2 in titanium reduced the impact strength appreciably, but his alloys contained appreciable amounts of oxygen, and it was difficult to attribute the embrittlement entirely to hydrogen.

The high temperature portion of the equilibrium diagram for the Ti-H system was established by McQuillan,[3] and his data generally corresponded with the results of Kirschfeld and Sieverts,[4] Gibb and Kruschwitz,[5] Bevington, Martin, and Mathews,[6] and other investigators. The lower concentration limit of the hydride phase varied considerably among investigators, and Gibb and Kruschwitz[5] had shown that small amounts of contaminants in either titanium or hydrogen caused considerable variation in this lower limit. In view of this pronounced effect of contaminants, the extrapolated value of McQuillan's, about 48 atomic pct at the 325°C eutectoid, appeared the most reasonable. McQuillan's lowest experimental value was 54 atomic pct at a temperature of 450°C.

The density of titanium had been shown to decrease with increasing hydrogen content, but discrepancies were noted among the various investigations. The best values indicated that the density was decreased from 4.56 grams per cc at 0.27 atomic pct[1] to 3.84 grams per cc at 61.3 atomic pct.[6]

This investigation was conducted to determine the effects of hydrogen on α titanium, both in high purity and commercially pure metals, and on high purity α titanium alloys.

Material and Fabrication

The literature indicated that impurities in either the hydrogen or the titanium must be kept to a minimum. For this reason, the initial studies were made with high purity iodide-refined titanium. The vendor's analysis of the iodide titanium and the hydrogen content of the as-fabricated ¼ in. diameter rods are shown in Table I. Two 1 lb ingots were prepared by arc melting under argon in a water-cooled crucible, using a water-cooled tungsten electrode. This equipment has been described.[7] As-cast Brinell hardness numbers of 88 and 58 for these ingots were low enough to insure that the high purity of the metal was maintained during arc melting. The ingots were hot forged to ¾ in. diameter rods at 800°C, cleaned by grit blasting, and then cold swaged to ½ in. diameter rods. The rod was then air annealed at 725°C, ground to remove scale, and cold swaged to ¼ in. diameter (75 pct reduction in area). The material was then annealed for 1 hr in argon at 800°C to develop an equiaxed α grain size of about 0.05 mm. Microtensile specimens and microimpact specimens were machined from this rod prior to treatment in the Sieverts-type absorption apparatus.

Two lots of commercially pure RC-55 titanium were also used in this investigation to determine the effects of hydrogen and the normal impurities on mechanical properties. The commercially pure RC-55 alloy was obtained in the form of ⅝ in. diameter rod. The analysis of this material is also given in Table I. The ⅝ in. diameter rods were hot swaged

G. A. LENNING is Principal Metallurgist, C. M. CRAIGHEAD is Assistant Chief, and R. I. JAFFEE, Member AIME, is Chief, Nonferrous Physical Metallurgy Div., Battelle Memorial Institute, Columbus, Ohio.

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Table I. Analysis of Materials

Element*	Composition, Pct	
	Lot IT-237	Lot IT-249
High Purity Iodide Titanium		
Mn	0.045	0.01
Fe	0.01	0.0035
Al	0.001	0.013
N	0.003	0.004
H_2†	0.009 ± 0.001	0.0072 ± 0.0007
Ti (by difference)	99.92	99.95

Element	Composition, Pct	
	RC-55-1	RC-55-2
Commercially Pure Titanium (⅝ In. Diameter Rod)		
C	0.03	0.02
N	0.03	0.03
Fe	0.27	0.31
H_2	0.0055 ± 0.0006 (0.26 ± 0.03 atomic pct)	0.0051 ± 0.0005 (0.24 ± 0.03 atomic pct)

* Less than 0.004 wt pct of Mo, Pb, Cu, Sn, Mg, and Ni were also noted.
† After melting and fabrication to ¼ in. diameter rod.

to ¼ in. diameter (84 pct reduction in area) at 700°C. Tensile and impact specimens were then machined from this rod, prior to treatment in the Sieverts apparatus.

A limited investigation was made of the effect of hydrogen on α titanium alloys containing nitrogen and aluminum as α stabilizers. The three alloys investigated had 0.06 and 0.18 wt pct N and 2.55 wt pct Al, respectively. These alloys were double arc melted to insure homogeneity. The ingots were then hot forged to ¾ in. rounds at 870°C, followed by swaging to ¼ in. diameter rod at 750°C.

The hydrogen used was commercially pure tank hydrogen which was purified by initial absorption in titanium turnings.

Hydrogenation and Vacuum Annealing

Hydrogenation and vacuum annealing were done in a modified Sieverts-type apparatus. The principal modification was in the purification of hydrogen by absorbing and desorbing the gas in heated titanium turnings. Hydrogen additions were measured from pressure readings on a system of known volume. A three-stage McLeod gage with a range from 10^{-4} to 12 mm of Hg was used for small additions, and larger additions were measured with a 3 mm diameter capillary manometer. Furnace tubes were made of Vycor and the specimens were supported in a molybdenum-wire basket or can so that only three-point contact occurred between the titanium specimens and the molybdenum.

Hydrogenation of the titanium specimens was carried out at temperatures from 820° to 880°C. The time required for absorption was of the order of 15 to 30 min at these temperatures. After the gas was absorbed, the specimens were furnace cooled to 680° or 720°C and equilibrated, generally for 1 hr, to insure homogeneity. Homogeneity in single specimens was established by microscopic examination and by vacuum-fusion analyses of layers machined from a ¼ in. diameter rod. The distribution of hydrogen noted microscopically was uniform throughout the cross section of the samples, and the analyses indicated that homogeneous specimens were obtained. Exceptions were observed in samples containing more than 15 atomic pct H_2, and these higher hydrogen specimens showed a thin hydride-rich surface layer. This was attributed to absorption of hydrogen during cooling of the specimens after the equilibration treatment.

Uniformity in hydrogen content between specimens hydrogenated in the same chamber also was determined. The results of vacuum-fusion analyses of two sets of duplicate specimens are shown in Table II. These results are in excellent agreement, indicating that a number of samples could be hydrogenated at the same time without appreciable variation in composition.

The apparatus and method used for vacuum-fusion analyses of the Ti-H alloys have been described.[5] Samples from 11 hydrogenation treatments were analyzed and, in all cases, the calculated addition was within the limit of accuracy of the analytical equipment.

All the titanium used in this work contained some hydrogen in the as-received condition. The amount varied from 0.005 to approximately 0.010 wt pct. In order to determine the properties of hydrogen-free material, it was necessary to remove the hydrogen by vacuum annealing. The vacuum-annealing treatments were made in the Sieverts apparatus in the temperature range of 800° to 870°C. The time of vacuum annealing varied according to the amount of material being outgassed. Generally, titanium which had been outgassed to a hot vacuum of 5×10^{-6} mm of Hg at 800° to 820°C had its hydrogen content reduced to the order of 0.001 wt pct.

Results

Constitution: The microstructures of representative high purity Ti-H alloys with less than 8 atomic pct H_2 have been presented previously.[5] In this range, titanium hydride precipitates at low temperatures into well-developed platelets or line markings if the rate of cooling is slow, or into a finely dispersed form in water-quenched specimens. The structures of representative higher hydrogen content alloys are shown in Figs. 1 to 3. Fig. 1 shows the structure of a high purity 25.1 atomic pct H_2 alloy as hydrogenated. The dark-etching constituent consists of transformed β (α and hydride), and the light-etching phase is α. Fig. 2 shows a high purity 40 atomic pct H_2 alloy as hydrogenated. This structure is close to the eutectoid composition, and the light-etching constituent is α. Fig. 3 shows the structure of a high purity 50 atomic pct H_2 alloy in the as-hydrogenated condition. The light-etching phase is hydride which has polished and etched in relief.

Fig. 4 shows an equilibrium diagram for the Ti-H system, taken chiefly from McQuillan[6] but with modifications which were indicated from the present investigation. The modifications were all in the low temperature portion of the diagram and are as follows: 1—addition of the curve showing the solubil-

Table II. Results of Vacuum-Fusion Analyses

Sample	Hydrogen Addition, Wt Pct	Hydrogen by Analysis, Wt Pct
C-81	0.0187	0.020 ± 0.002
C-91	0.0187	0.018 ± 0.002
C-131	0.117	0.11 ± 0.01
C-141	0.117	0.12 ± 0.01

Fig. 1—Iodide titanium-25.1 atomic pct H_2, as hydrogenated. Structure shown is α + transformed β. Note hydride phase precipitated from α on cooling. X250. Area reduced approximately 50 pct for reproduction.

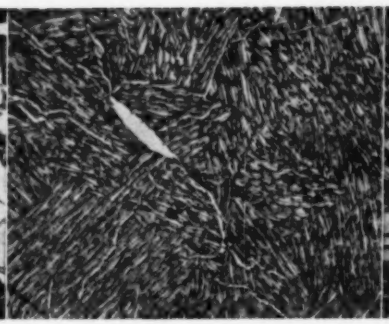

Fig. 2—Iodide titanium-40 atomic pct H_2, as hydrogenated. Structure shown is α + transformed β (α + γ). X250. Area reduced approximately 50 pct for reproduction.

Fig. 3—Iodide titanium-50 atomic pct H_2, as hydrogenated. Structure shown is hydride + transformed β (α + γ). X250. Area reduced approximately 50 pct for reproduction.

ity limit of hydrogen in α titanium, 2—inclusion of two eutectoid horizontals to represent the hysteresis observed in thermal analysis and dilatometer studies, 3—a shift of the eutectoid composition to higher hydrogen concentrations, and 4—an increase in the hydrogen concentration of the lower limit of the hydride phase. These four alterations are discussed in detail below.

The low room-temperature solubility of hydrogen in high purity α titanium has already been pointed out.[9] In view of the pronounced effect of hydrogen on the impact strength of titanium, it was considered desirable to determine the solid-solubility curve for hydrogen in α. This investigation was made microscopically over a range of hydrogen concentration from 0.05 to 8.4 atomic pct. Specimens were solution treated for 24 or 48 hr in argon or air at 400°C to insure solution of the hydride, slowly cooled to the desired temperature, held 2 hr at temperature, and then water quenched. In examining the specimens, it was necessary to use the criterion of hydride precipitation into massive Widmanstaetten plates, since the hydride could not be retained in solution by water quenching. This is illustrated for a 1.2 atomic pct H_2 alloy in Figs. 5 and 6. The structure in Fig. 5 was obtained by quenching from the solution range, and the hydride has precipitated as a finely dispersed phase, either on quenching or by aging during preparation for metallographic examination.

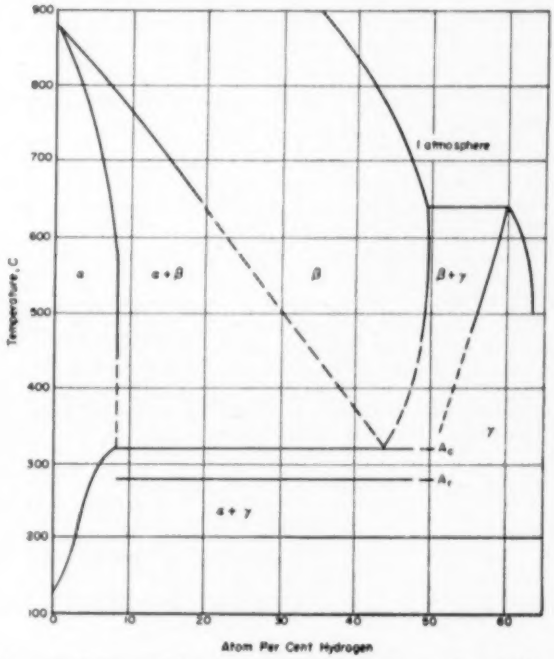

Fig. 4—Ti-H equilibrium diagram based on the results of McQuillan and the present investigation.

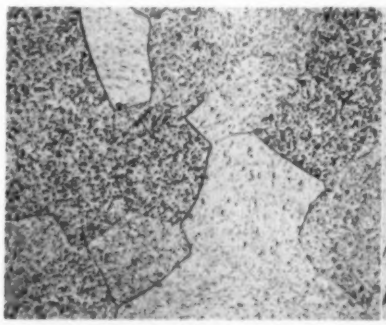

Fig. 5—Ti-1.2 atomic pct H_2 alloy. Heat treated for 24 hr at 400°C in argon, furnace cooled to 150°C, held 2 hr at 150°C, and quenched in cold water. X250. Area reduced approximately 50 pct for reproduction.

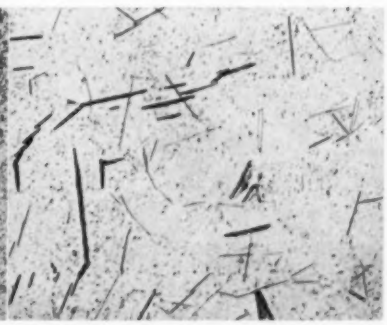

Fig. 6—Ti-1.2 atomic pct H_2 alloy. Heat treated for 24 hr at 400°C in argon, furnace cooled to 125°C, held 2 hr at 125°C, and quenched in cold water. X250. Area reduced approximately 50 pct for reproduction.

All specimens were prepared by mounting in a plastic that set at room temperature. The structure in Fig. 6 was obtained by quenching from the two-phase region and clearly shows precipitation of the massive hydride. In all cases, the precipitation of massive hydride was quite pronounced and justified the use of this criterion.

The solubility of hydrogen below 125°C is between 0.001 and 0.0029 wt pct H_2 (0.05 and 0.14 atomic pct). An 8.4 atomic pct H_2 alloy contained a small amount of transformed β, confirming McQuillan's extrapolated solubility limit of 8 atomic pct for hydrogen in α titanium at the eutectoid temperature. Fig. 7a shows the α solubility curve of hydrogen in high purity titanium and the experimental points on which the curve is based.

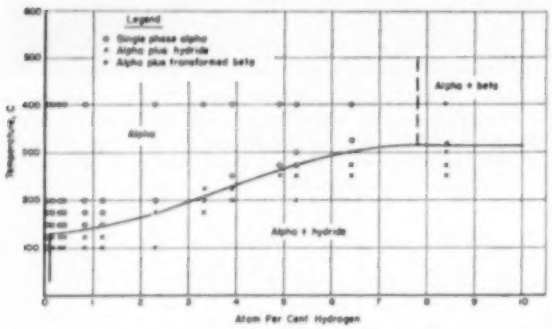

Fig. 7a—Low temperature α solubility limit for hydrogen in high purity titanium.

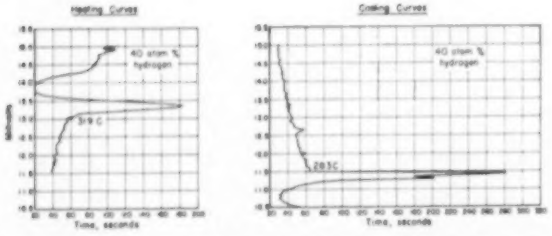

Fig. 7b—Inverse-rate heating and cooling curves for a 40 atomic pct H_2 alloy.

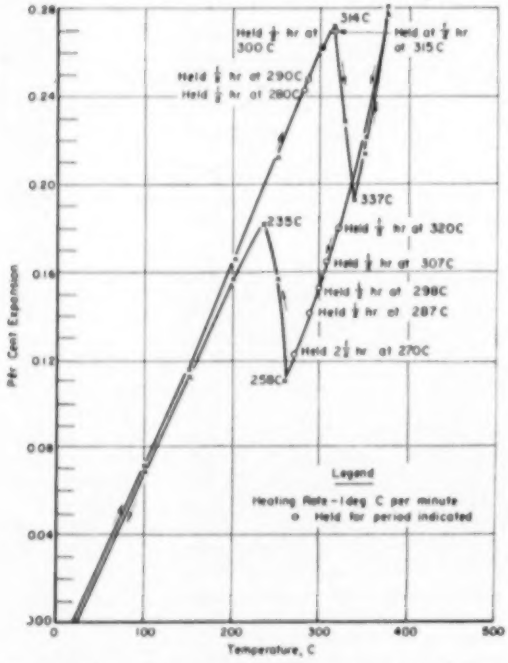

Fig. 7c—Dilatometer curves for a 20 atomic pct H_2 alloy showing hysteresis at the eutectoid transformation.

The double-eutectoid horizontal shown in Fig. 4 illustrates the hysteresis observed by thermal analysis from heating and cooling curves made on alloys with 25.1, 32, and 40 atomic pct H_2 and by a dilatometer investigation of a 20 atomic pct H_2 alloy. The heating and cooling curves were made on small specimens at heating and cooling rates of approximately 1°C per min. The average temperature of arrest on heating was 319°C and on cooling, 281°C, or a hysteresis of 38°C. The inverse-rate method was employed, and the breaks were quite prominent and reproducible, as shown in Fig. 7b. The dilatation investigation was conducted with an autographic quartz dilatometer and showed contraction breaks at 312°C on heating and expansion breaks at 259°C on cooling. Dilatation data are based on an average of four complete cycles between room temperature and 400°C. Fig. 7c shows typical dilatometer data for the 20 atomic pct H_2 alloy. During the last dilatometer cycle, the temperature was held constant for 30 min at temperatures of 280°, 290°, and 300°C on heating without affecting the arrest. Similarly, holding the temperature constant for 30 min at 320° and 307°C, 1½ hr at 298°C, 1 hr at 287°C, and 2½ hr at 270°C during cooling did not shift the temperature of arrest.

Three characteristics of a martensite transformation were indicated from this investigation of the eutectoid transformation. First, the β decomposition could not be suppressed by water quenching to room temperature. Second, the transformation temperature was not affected by decreasing the heating and cooling rate. Third, the reversibility of the transformation is associated with an appreciable hysteresis.

Other characteristics observed for the eutectoid transformation indicated that it did not occur by the martensite reaction but by a diffusion-controlled reaction involving a highly mobile solute atom. No acicular needles were observed in the structures quenched from above the eutectoid temperature. This is shown in Fig. 8 for a 50 atomic pct H_2 alloy annealed at 450°C for 7 hr and water quenched. The eutectoid structure shown is somewhat finer than for the slowly cooled materials, Fig. 3, but the struc-

Fig. 8—High purity Ti-50 atomic pct H_2 alloy as air annealed at 450°C for 7 hr and water quenched. ×250. Area reduced approximately 50 pct for reproduction.

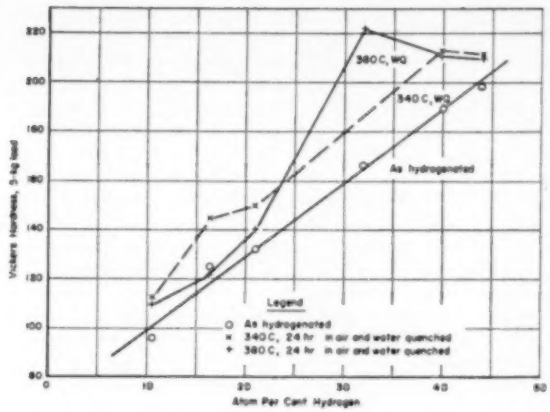
380 C, WQ
340 C, WQ
As hydrogenated
Legend
As hydrogenated
340 C, 24 hr in air and water quenched
380 C, 24 hr in air and water quenched
Vickers Hardness, 5-kg load
Atom Per Cent Hydrogen

Fig. 9—Effect of quenching from above the eutectoid temperature on hardness of high purity Ti-H alloys.

ture is not typically martensitic. Vickers hardness measurements taken on alloys quenched from above the eutectoid temperature were not appreciably higher than for slowly cooled alloys, as shown in Fig. 9, and this would also support the diffusion mechanism. However, the considerable hysteresis observed in connection with this reaction would be difficult to explain on the basis of a diffusion mechanism. Additional study of this reaction would be desirable before drawing definite conclusions.

The shift in eutectoid composition to a higher hydrogen concentration than that shown in the extrapolated portion of McQuillan's diagram was indicated by the microstructure of a 43.8 atomic pct H_2 alloy. This structure as slow cooled is shown in Fig. 10 and consisted of α (light) and transformed β (dark). This alloy contained only a small amount of α, less than the relative amount indicated in the micrograph, and therefore the eutectoid composition is thought to be close to 44 atomic pct H_2.

An increase in the hydrogen content of the lower limit of the hydride phase was also indicated by this investigation. Fig. 3 shows the microstructure of the 50 atomic pct H_2 alloy as hydrogenated. This structure consisted of transformed β and hydride. Fig. 11 shows the structure of this alloy annealed in air at 300°C for 7 hr and water quenched. This structure also consisted of transformed β and hydride The amount of transformed β was also relatively large. McQuillan indicated that the 50 atomic pct H_2 alloy was all γ phase (hydride) at 300°C. Annealing in air in this temperature range has not resulted in a loss of hydrogen, because of the low temperature and the protection afforded by the oxide scale. These structures indicated that the lower limit of the γ hydride phase at both 300°C and room temperature was above 50 atomic pct H_2. This observation would be in accord with the shift of McQuillan's value for the eutectoid to higher hydrogen concentrations, shown in Fig. 4.

The marked decrease in hydrogen solubility in the α phase between 300° and 125°C has led to specu-

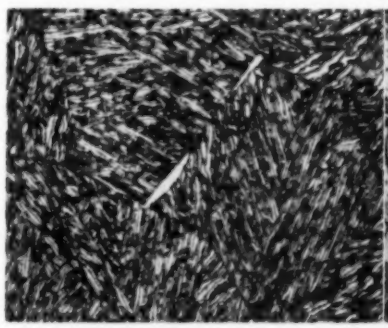

Fig. 10—Iodide titanium-43.8 atomic pct H_2. Structure shown is α + transformed β (α + γ). X250. Area reduced approximately 50 pct for reproduction.

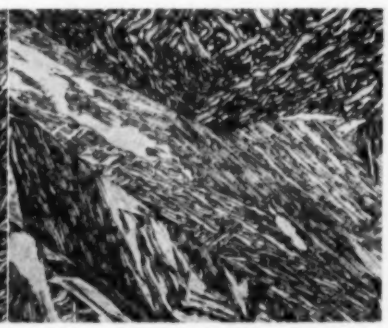

Fig. 11—Iodide titanium-50 atomic pct H_2 annealed at 300°C for 7 hr and water quenched. Structure shown is hydride + transformed β. X250. Area reduced approximately 50 pct for reproduction.

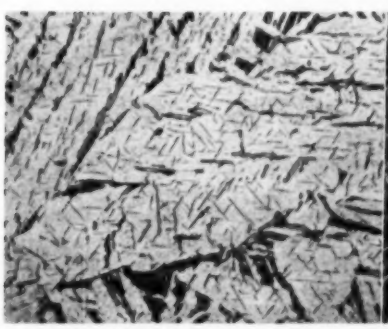

Fig. 12—Iodide titanium-8.4 atomic pct H_2 alloy annealed at 100°C for 19 days. Structure consists of α and hydride. X250. Area reduced approximately 50 pct for reproduction.

Fig. 13—Iodide titanium-32 atomic pct H_2 alloy annealed at 100°C for 19 days. Structure consists of α + β. X250. Area reduced approximately 50 pct for reproduction.

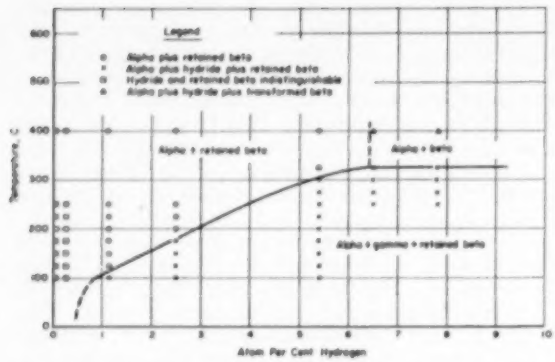
Legend
Alpha plus retained beta
Alpha plus hydride plus retained beta
Hydride and retained beta indistinguishable
Alpha plus hydride plus transformed beta
Alpha + retained beta
Alpha + beta
Alpha + gamma + retained beta
Temperature, °C
Atom Per Cent Hydrogen

Fig. 14—Low temperature solubility relation for hydrogen in commercial (RC-55) titanium containing about 0.3 pct Fe.

effects of the impurities (oxygen, nitrogen, and iron) in the commercial material were to decrease the α solubility of hydrogen at the eutectoid temperature and to cause an apparent increase in solubility at room temperature. The retained β phase in this material, resulting from the iron impurity of about 0.3 wt pct (RC-55-1, 0.27 pct Fe), caused some uncertainty in establishing the room-temperature solubility limit as indicated by the dashed line in Fig. 14. This difficulty in distinguishing metallographically between small amounts of hydride and retained β is illustrated in Figs. 15 and 16a, where the structures of 0.05 and 0.26 atomic pct H_2 specimens are shown. The 0.05 atomic pct sample was annealed at 400°C for 24 hr and furnace cooled. The structure consists of α plus retained β, stabilized by the iron impurity. The 0.26 atomic pct sample was treated for 63 hr, at 100°C, and oven cooled. If the

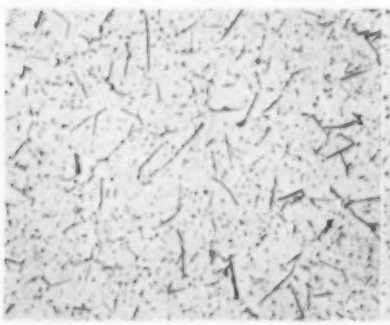

Fig. 15—Commercial titanium with 0.3 wt pct Fe and 0.05 atomic pct H_2 annealed at 400°C for 24 hr and furnace cooled. Structure consists of α + retained β. X250. Area reduced approximately 50 pct for reproduction.

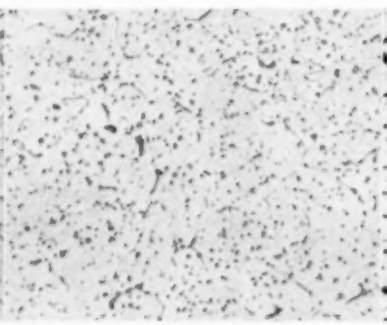

Fig. 16a—Commercial titanium with 0.3 wt pct Fe and 0.26 atomic pct H_2 annealed at 100°C for 63 hr and oven cooled. Structure consists of α + retained β. X250. Area reduced approximately 50 pct for reproduction.

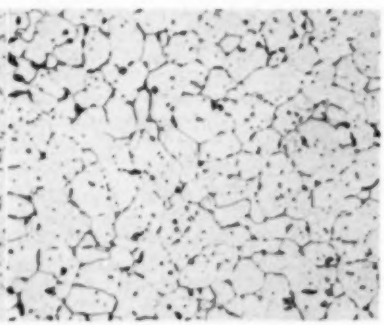

Fig. 16b—Commercial titanium with 0.3 wt pct Fe and 1.1 atomic pct H_2 annealed at 400°C for 24 hr, furnace cooled to 100°C, held 3 hr, and water quenched. Structure consists of α + retained β + hydride. X250. Area reduced approximately 50 pct for reproduction.

lation that a lower hydride of titanium than γ might be formed by a peritectoid reaction at 125°C. To investigate this possibility, specimens from an 8.4 and 32 atomic pct H_2 alloy were aged at 100°C for 19 days. Figs. 12 and 13 show the structures resulting from this treatment. These microconstituents were identified metallographically as α and hydride, and no evidence of a lower hydride was indicated.

The low temperature solubility relation for hydrogen in commercial titanium (RC-55) was also investigated. The procedure used in this investigation closely paralleled that for high purity titanium, and the results are shown in Fig. 14. The overall

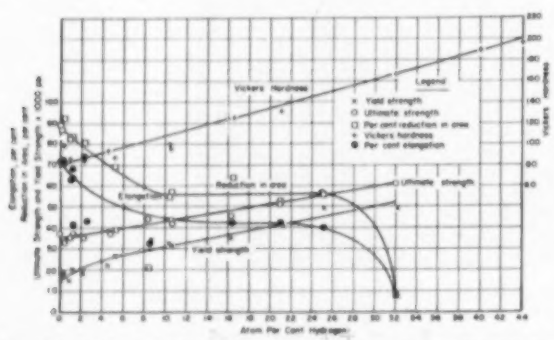

Fig. 17—Vickers hardness and tensile properties of high purity Ti-H alloys.

hydride were not in solution, the structure should consist of α plus hydride plus retained β, and the hydride would be expected to be present as platelets. However, comparison of the two structures shows no difference in the amount of second phase, and there are no constituents, definitely identified as hydride, in either of the specimens. Repeated attempts at long-time, low temperature aging treatments and very slow cooling of these two alloys failed to produce any microconstituent which could definitely be identified as hydride. Fig. 16b for the same base with 1.1 atomic pct H_2 clearly shows hydride platelets in the matrix of α and retained β.

Mechanical and Physical Properties: Tensile data were obtained for all materials by using specimens 2⅛ in. long, with a reduced section of ⅛ in. diameter and ¾ in. length. This specimen is a geometrical reduction of the 2 in. gage length, standard ASM test specimen, and its specifications were taken from the 1948 edition of the ASM *Metals Handbook*. Information obtained from these tests included ultimate strength, 0.2 pct offset yield strength, percentage of elongation in 0.5 in., and percentage of reduction in area. Conventional SR-4 resistance-type gages were used over the elastic region.

Fig. 17 is a plot of ultimate strength, 0.2 pct offset yield strength, percentage of reduction in area, percentage of elongation in ½ in., and 5 kg Vickers hardness vs hydrogen concentration for high purity Ti-H alloys in the as-hydrogenated condition.

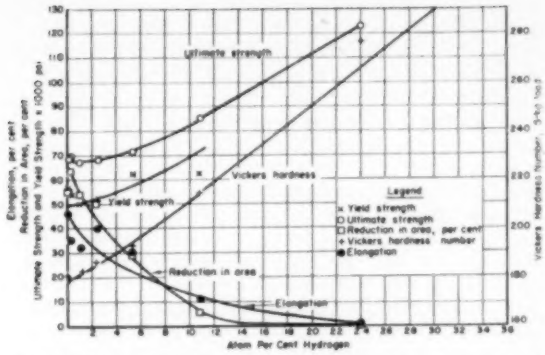

Fig. 18—Vickers hardness and tensile properties of commercial Ti-H alloys.

Strength properties and hardness show a gradual increase with increasing hydrogen content. Reduction in area and elongation decrease between 0 and 10 atomic pct H_2 but then remain about constant to a concentration of 25 atomic pct. At hydrogen concentrations above 25 atomic pct the ductility drops off sharply. A 44 atomic pct H_2 sample displayed no ductility, and the specimen broke in handling before it could be tested.

The tensile properties of commercial (RC-55) Ti-H alloys are shown in Fig. 18. Ultimate strength, yield strength, and Vickers hardness show an increase with increasing hydrogen contents. The ductility, as indicated by reduction in area and elongation, drops to a very low value beyond 10 atomic pct. Comparison of the properties of high purity and commercially pure alloys shows that the effect of impurities in the commercial material is most pronounced on tensile ductility, low values being found with 10 atomic pct H_2 compared to 25 atomic pct for the high purity base.

Impact properties of the Ti-H alloys were obtained by using a 0.225 in. diameter, 1⅛ in. long, cylindrical Izod specimen having a 45° V-notch with a radius of 0.005 in. and a root diameter of 0.150 in. This specimen has been illustrated in other work.[10] Magnitude of energy absorbed and transition temperatures measured with this specimen correlate quite well with the same properties as measured by using the standard Charpy V-notch specimen. This correlation was made for a number of materials, and a conversion factor of 0.8 was obtained for converting inch-pounds obtained on the Izod specimen to Charpy V-notch foot-pound values.

Impact strengths of high purity Ti-H alloys were determined at various temperatures from −196° to 100°C for alloys in the slowly cooled condition. The impact strength of several high purity Ti-H alloys is shown as a function of testing temperature in Fig. 19. The specimens containing 0.05 and 0.14 atomic pct H_2 did not fracture completely under a load of 100 in.-lb, even at −196°C. At room temperature, the material with hydrogen contents of 0.3 and 0.4 atomic pct fractured with an absorption of energy of 43 and 22 in.-lb respectively. At hydrogen concen-

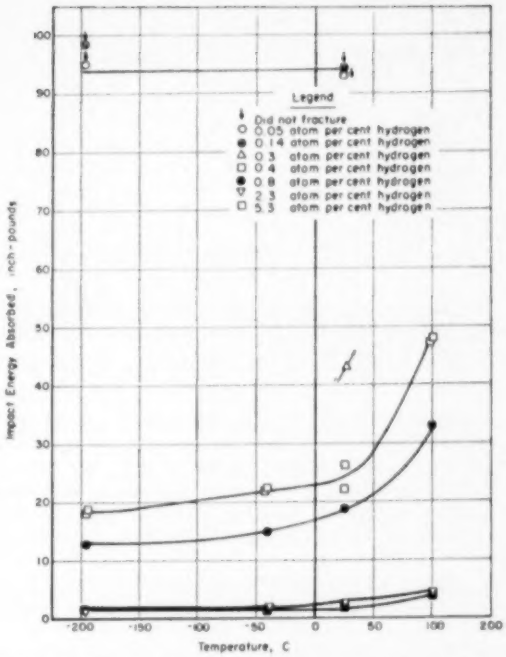
Impact Energy Absorbed, inch-pounds
Legend
Did not fracture
0.05 atom per cent hydrogen
0.14 atom per cent hydrogen
0.3 atom per cent hydrogen
0.4 atom per cent hydrogen
0.8 atom per cent hydrogen
2.3 atom per cent hydrogen
5.3 atom per cent hydrogen
Temperature, C

Fig. 19—Effect of hydrogen on impact properties of high purity titanium. Specimens cooled through hydride-precipitation range at about 15° to 20°C per min.

Table III. Impact Properties of Commercially Pure Ti-H Alloys

Hydrogen Content, Atomic Pct	Impact Energy Absorbed at Indicated Temperatures, In.-Lb			
	−196°C	−40°C	Room Temperature	100°C
0.05	39	38	36	39
0.26	16.5	21	24	32.5
1.1	4.5	6.0	11	20.4
2.5	0.5	1.0	1.0	3.0

Table IV. Tensile Properties, Hardnesses, and Impact Strengths of High Purity Alpha Titanium Alloys at Two Hydrogen Levels

Alloy Addition, Wt Pct	Hydrogen		Treatment	Tensile Strength, Psi	0.2 Pct Offset Yield Strength, Psi	Elongation, Pct	Reduction in Area, Pct	Room-Temperature Impact Strength, In.-Lb
	Atomic Pct	Wt Pct						
0.06 N	0.07	0.0014	Vacuum annealed 775°C 2 hr, furnace cooled	82,000	72,000	36	55	24
0.06 N	0.63	0.0133	700°C 1 hr in argon, furnace cooled	77,000	63,000	30	57	4
0.18 N	0.09	0.0018	Vacuum annealed 775°C 2 hr, furnace cooled	114,000	110,000	23	26	16
0.18 N	0.6	0.0127	700°C 1 hr in argon, furnace cooled	122,000	117,000	25	23	3
2.55 Al	0.07	0.0015	Vacuum annealed 775°C 2 hr, furnace cooled	93,000	78,000	26	47	41
2.55 Al	0.6	0.0129	900°C 1 hr in argon, furnace cooled	96,000	82,000	23	39	7

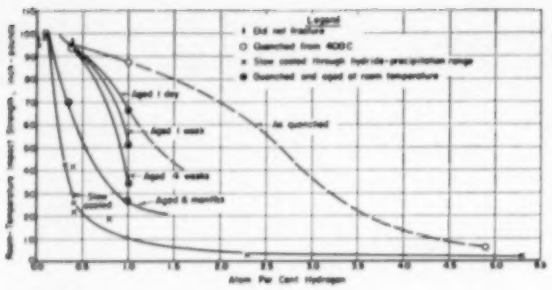

Fig. 20—Beneficial effect on toughness of water quenching from α phase field and toughness decay on room temperature aging for high purity Ti-H alloys.

trations above 0.8 atomic pct, the impact strength is quite low. The impact-temperature curves are most interesting. Comparatively little change in impact occurs from −196°C to room temperature. Increasing hydrogen contents appear chiefly to lower the level of impact-energy absorption over this temperature.

The impact strengths of commercially pure Ti-H alloys at the various testing temperatures are shown in Table III.

The effects of hydrogen on the impact strength of commercial and high purity titanium were similar, except that the impact strength of hydrogen-free, commercial titanium was lower than that for hydrogen-free, high purity titanium. Again, increasing hydrogen content progressively lowered the general level of impact-energy absorption at each testing temperature.

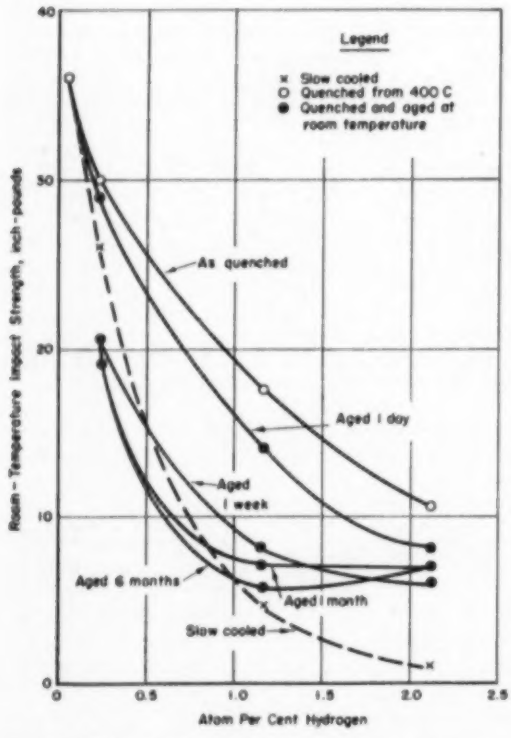

Fig. 21—Effect of solution treatment and room temperature aging of commercial RC-55 Ti-H alloys.

The effect of hydrogen on the α stabilized alloys containing nitrogen and aluminum was similar to that on high purity unalloyed titanium, as shown in Table IV. Tensile strength, yield strength, elongation, and reduction in area were not significantly affected, but the room-temperature impact strength was considerably improved by the lower hydrogen contents found after vacuum annealing. The microstructures of the vacuum-annealed specimens were free of hydride, but the structures of the alloys with the as-fabricated hydrogen contents indicated in Table IV showed an appreciable amount of well-defined hydride.

An investigation of a hydrogen solution treatment and quench was made to determine the effect on impact strength. Also, the effect of room-temperature aging was investigated. High purity Ti-H alloys with 0.4, 1.0, and 4.9 atomic pct H_2 were solution

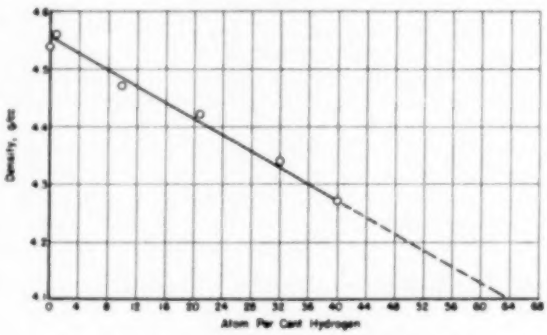

Fig. 22—Density of high purity base Ti-H alloys.

treated for 64 hr at 400°C in argon and quenched in cold water. The results of this investigation are shown in Fig. 20. The impact strengths of the 0.4 and 1.0 atomic pct alloys were markedly improved by the solution treatment. The cause for this improvement is evident from the microstructures of the alloys. Slow-cooled, as-hydrogenated alloys showed relatively large hydride plates, whereas alloys quenched from the α solid-solution temperature range showed a finely dispersed hydride phase. The improvement in the 4.9 atomic pct H_2 alloy was slight because of the low initial energy-absorption values at the higher hydrogen content. Preliminary aging studies of solution-treated alloys at 100°C had shown a tendency for the hydride to agglomerate into coarser particles, and it was expected that room-temperature aging might also agglomerate the hydride, with a deleterious effect on impact strength. After six months' aging at room temperature, the 0.4 atomic pct H_2 material still showed a high impact strength. However, the 1.0 atomic pct alloy showed a marked decrease in strength from the solution-treated level after only one day of room-temperature aging. It would appear that any permanent beneficial effects from solution treatment could be realized only with alloys containing less than 0.5 atomic pct H_2. However, even here, aging at temperatures slightly above room temperature would be expected to accelerate markedly the decay of the enhanced toughness.

The effect of solution treatment and aging on the impact strength of commercial purity base alloys was also investigated. These alloys contained 0.24, 1.2, and 2.2 atomic pct H_2, and the same solution

treatment was used. The results are shown in Fig. 21. In general, the same type of decay was noted for the alloys with 1.2 and 2.2 atomic pct H_2. A somewhat different behavior is noted for the 0.24 atomic pct H_2 alloy. Only a small improvement over the slow-cooled impact value was noted from solution treatment of this alloy. This might be expected from the indications that most of the residual hydrogen, in alloys with iron present as an impurity, is dissolved in the retained β phase at room temperature. However, after one week's aging, the impact strength of the solution-treated, 0.24 atomic pct material was below that for the slowly cooled material. This behavior would not be expected on the basis of the solution-treatment and aging studies of the high purity alloys. One explanation of the behavior of the solution-treated 0.24 atomic pct H_2 alloy would be that, at 400°C, some of the hydrogen is dissolved in the α, although most would be in the β because of higher solubility. Room-temperature aging of the quenched alloy might precipitate hydride from the α, thereby reducing the impact strength. Slow cooling, conversely, might allow the hydrogen to enter into solution in the retained β phase and not reduce the impact values so markedly.

X-ray diffraction patterns of the hydride phase in a high purity 43.8 atomic pct H_2 alloy indicated that this phase is face-centered cubic. The lattice parameter obtained, 4.40 ± 0.02Å, is in agreement with McQuillan's reported value of 4.41Å.[8] Densities of alloys with 0.05, 0.9, 10.6, 21, 32, and 40 atomic pct H_2 were determined by the method of displacement in water. These densities are plotted against hydrogen content in Fig. 22. Generally, the density values determined in this investigation were slightly higher than those reported in the literature.[1, 4, 8]

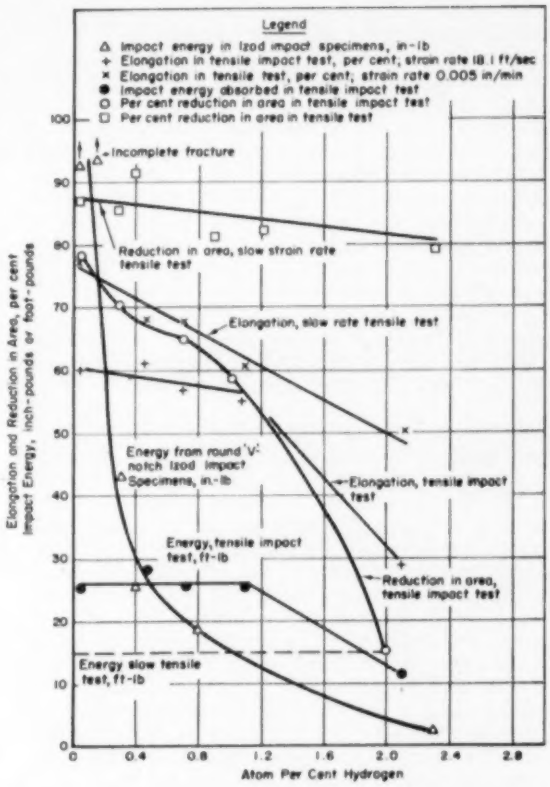

Fig. 23—Effect of strain rate on reduction in area, elongation, and breaking energy in tensile tests at various hydrogen levels and the effect of hydrogen on the energy absorbed in the Izod impact test.

Discussion

Hydrogen contamination of titanium is generally less than 200 ppm (about 1 atomic pct). The effect of this amount of hydrogen on tensile properties is negligible, but the effect on notch-bar impact properties is profound. It is pertinent to inquire as to the reason for this set of circumstances. The presence of Widmanstaetten plates of a brittle hydride phase might be expected to have an adverse effect on ductility in general, but not necessarily only one type of ductility.

This situation would indicate that hydrogen either increases the strain-rate sensitivity or the notch sensitivity of titanium. In order to determine which of the two effects predominated, notched Charpy impact specimens of commercial Ti-H alloys with 0.05, 0.26, and 2.0 atomic pct H_2 were prepared for testing at impact speeds and at slow speeds. The impact tests were made at a strain rate of 18.1 ft per sec in a conventional Riehle impact tester. The slow-bend tests were made at a strain rate of 0.08 in. per min. The anvil and strikers for the two strain rates had similar dimensions. The results of this investigation are given in Table V. At both strain rates, the level of absorbed energy was about the same for a given hydrogen content, which would indicate a lack of strain-rate sensitivity, and that notch sensitivity was controlling.

Table V. Results of Impact and Slow Bend Tests on Ti-H Alloys

Hydrogen Content, Atomic Pct	Energy Absorbed in Breaking, Ft-Lb: Impact Tests, 18.1 Ft per Sec	Energy Absorbed in Breaking, Ft-Lb: Slow Bend Tests, 0.08 In. per Min
0.05	30	15.5
0.05	32	20
0.26	19.5	18.5
0.26	20	22
2.0	3.5	4.0
2.0	—	6.5

Additional work to determine the effect of strain rate on mechanical properties was done, using the standard microtensile specimens. Duplicate tensile specimens of high purity titanium were prepared with 0.05, 0.47, 0.71, 1.1, and 2.1 atomic pct H_2. One tensile specimen for each hydrogen concentration was tested at a strain rate of 0.005 in. per min, and the other was tested under tensile-impact conditions in a modified Riehle impact tester at a strain rate of 18.1 ft per sec. The results of this investigation are shown in Fig. 23, where breaking energy, elongation, and reduction in area are plotted against hydrogen content. The room-temperature impact strength of V-notch, Izod impact, bend specimens is shown also. The elongation, reduction in area, and breaking energy in tension were not markedly decreased at hydrogen contents through 1.1 atomic pct for either strain rate. A decrease in elongation, reduction in area, and breaking energy did occur at 2.1 atomic pct H_2. However, the embrittlement in tensile impact does not occur at such low hydrogen contents as it does in the V-notch Izod impact specimens. This indicates that the embrittlement in-

duced by hydrogen is not primarily the result of a detrimental effect on strain-rate sensitivity.

The hydrogen content of most commercial titanium so far tested has been around 50 ppm and, although 25 ppm or less is desirable, 50 ppm is not very damaging. In the presence of more than 50 ppm of hydrogen, vacuum annealing is a positive solution to the hydrogen problem. However, the kinetics of removal of hydrogen from large masses is not known, nor is the overall practicability of vacuum annealing known yet. Cooper and Bounds[11] have shown that vacuum annealing can be used successfully as a commercial operation on relatively thin-walled titanium tubing, so the operation is certainly feasible. Also, in the authors' experience, hydrogen is successfully removed from ½ in. diameter bar stock by a few hours in vacuum at about 850°C.

The relatively small difference between the impact strength of vacuum-annealed and 0.26 atomic pct (50 ppm) commercial titanium as compared to the sharp decrease in impact strength for high purity titanium in this hydrogen range would indicate that either the hydride phase did not precipitate in the commercial material or that there was a redistribution of this hydride. The absence of hydride from the microstructure of even slowly cooled commercial titanium with 0.26 atomic pct H_2 would indicate that the hydrogen was in solution in the globular retained β phase. Since the retained β phase results from the iron impurity, it would be expected that the room-temperature solubility of hydrogen in commercial titanium would vary according to the amount of iron impurity.

The apparent, relatively high solubility of hydrogen in retained β suggests the use of retained β as a hydrogen getter. However, this practice could not be generally recommended, since the β phase would be expected to become saturated with hydrogen and also to become brittle. When the β phase is present as intragranular globules, the effect of hydrogen on impact strength would probably not be too great, but when β is intergranular, as it often is at higher iron contents, a serious loss of ductility might occur.

A solution treatment of titanium containing hydrogen makes a definite improvement in toughness, compared to the slow-cooled condition. However, aging even at room temperature causes a decay in the enhanced properties. Aging at slightly elevated temperature would be expected to increase the rate of decay. Hence, heat treatment cannot be considered as a solution to the problem of poor toughness caused by hydrogen.

Summary

1—The solubility of hydrogen in α titanium decreases from about 8 atomic pct at 300°C to about 0.1 atomic pct at room temperature, the decrease in solubility being greatest down to 125°C. The hydride precipitates as plates at slow cooling rates and as a fine dispersion after quenching.

2—The eutectoid composition has been placed at about 44 atomic pct, and the lower limit for the range of homogeneity of the face-centered cubic hydride phase, γ, has been placed at slightly higher than 50 atomic pct.

3—The β eutectoid reaction has a hysteresis of about 40°C, which does not appear dependent on heating or cooling rate. Structures of transformed β are typically eutectoid, and little hardening is associated with the transformation, even after water quenching.

4—The effect of hydrogen on the mechanical properties of α titanium, including high purity iodide titanium, commercial titanium, and α alloys, is to decrease notch toughness without affecting tensile properties.

5—The cause for the effects of hydrogen on mechanical properties appears to be increased sensitivity to notches. Strain rate has no deleterious effects until higher hydrogen contents are reached.

6—The detrimental effects of hydrogen on notch toughness can be alleviated by vacuum annealing to remove hydrogen or by quenching through the precipitation range. The latter case gives a finely dispersed structure with enhanced toughness. Room-temperature aging causes a subsequent decay in toughness, and heat treatment cannot be considered as a remedy for the detrimental effects of hydrogen.

7—The presence of iron as an impurity in commercial titanium stabilizes a small amount of retained β phase to room temperature. Because hydrogen has a high solubility in β, the room-temperature solubility of hydrogen in commercial titanium is higher than in high purity titanium. This effect does not, apparently, eliminate the detrimental effects of hydrogen on notch toughness, perhaps because of embrittlement of the β phase.

Acknowledgment

The authors acknowledge the support of Watertown Arsenal, under whose technical supervision the work was conducted, and the helpful suggestions and discussions of Dr. L. D. Jaffe and Mr. D. C. Buffum of that Laboratory. Thanks are also due the authors' coworkers at Battelle: Mr. R. D. Buchheit for his work on metallography; Mr. H. W. Deem for dilatometric studies; and Dr. C. M. Schwartz for X-ray diffraction work.

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Load-Temperature History of Lattice Strain In Aluminum Alloy

by D. Rosenthal and M. Kaufman

IT would be of great importance to our understanding of the phenomena of fracture in metals if a unique relationship could be established between stress and some easily measurable parameter of deformation up to the fracture. It is known that no such relationship has been found when the parameter was the mechanically measured strain,[1] the major part of which is plastic. The present investigation was initiated to determine, among other things, if the X-ray or lattice strain* might be a more suitable parameter. To this end, annealed stress free** specimens of 61S aluminum alloy were tested at room temperature and liquid nitrogen temperature (−195°C), by first maintaining the same temperature from the beginning until the end of the test, and then with crossovers from one temperature to the other.

* For the definition of lattice strain see ref. 2.

** Freedom of stress in this particular case means absence of those stresses which can be checked by the technique employed.

Three major factors had to be taken into consideration in designing the specimens and testing equipment. The test setup must allow X-ray pictures to be taken while the specimen is under load (for as much as 2 hr at a given load). Provision must be made for maintaining the test specimen at the temperature of liquid nitrogen as well as at room temperature. In addition, the specimen must be able to rotate to allow the X-ray beam to take in as large a sample of grains as possible, to maintain centering, and to correct for effects of large grains.

The final design of the specimen and equipment is shown in Figs. 1 and 2. The specimen and grips are hollow, enabling liquid nitrogen to be poured in through a funnel-container arrangement while the specimen is rotating. The diameter of the specimens at the 2 in. gage section is 5/16 in. with a 3/16 in. diameter hole through the center. A small pilot valve in the lower grip allows a very small stream of liquid nitrogen to escape, thus assuring circulation of the liquid at all times. A low temperature silicone grease-tissue paper packing is used to seal all joints. The specimen is held in the grips by a pin. Spherical socket joints at top and bottom are used to allow for proper alignment. The grips are connected to the thrust bearings by means of plastic couplings to decrease the heat flow. Rotation is accomplished by means of a motor which drives a plastic gear (integral with the lower coupling) through a semiuniversal joint to allow for deformation of the specimen. The torque applied to the specimen is very small. The maximum stress caused by this torque at the highest loads reached was found to be less than 200 psi. The whole unit was mounted in a 10,000 lb testing machine. An insulation cage was placed around the specimen to cut down air circulation, and consequent frosting of the specimen, and also to reduce radiation and convection heat losses. A narrow window, covered on both sides with a thin plastic material allows the X-ray beam to reach the specimen. Thermocouples placed just outside the pilot hole at the lower grip, in the bottom of the funnel container and near the top of the funnel container permitted checks of the

Fig. 1—Hollow aluminum specimen.

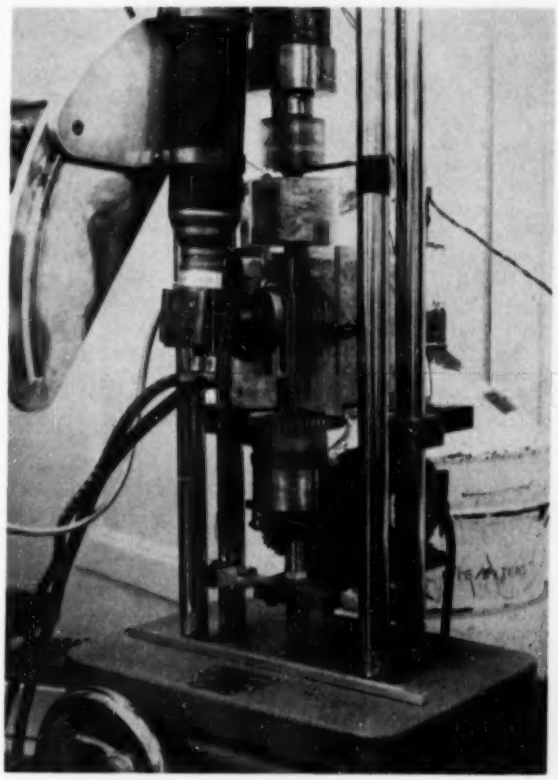
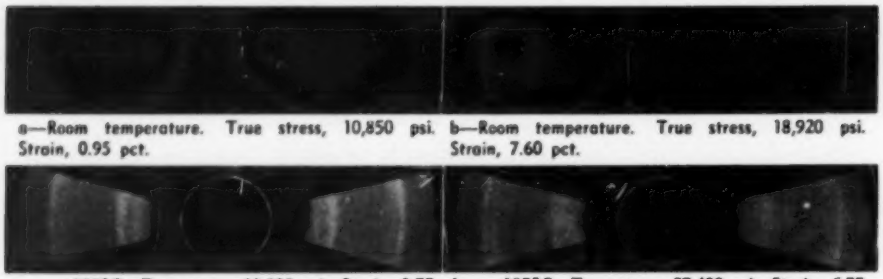


Fig. 2—Testing equipment, set up for −195°C test.

D. ROSENTHAL and M. KAUFMAN are associated with the Dept. of Engineering, University of California, Los Angeles.

Discussion on this paper, TP 3727E, may be sent, 2 copies, to AIME by May 1, 1954. Manuscript, April 1, 1953.

The work reported was sponsored partially by NOTS, Inyokern.



a—Room temperature. True stress, 10,850 psi. Strain, 0.95 pct.

b—Room temperature. True stress, 18,920 psi. Strain, 7.60 pct.

c— —195°C. True stress, 11,900 psi. Strain, 0.75 pct.

d— —195°C. True stress, 27,490 psi. Strain, 6.55 pct.

Fig. 3—Typical X-ray photograms. Inner doublet is that of 61S aluminum. Outer doublet is that of tungsten reference powder.

level of the liquid nitrogen. Trial tests showed that as long as the specimen was full of liquid nitrogen, the outer surface of the specimen remained within 1°C of the temperature of the liquid nitrogen. For strains within the elastic limit, SR-4 strain gages were used.† The device used to measure extension beyond the elastic limit is merely a "feeler" gage reading to 0.0001 in. fastened to one leg of an inside caliper, with the feeler as one of the contact points. Extension is measured by placing the contact points between the collars of the specimen and noting the reading on the feeler gage. The distance between the contact points is then measured on a suitable micrometer by adjusting the micrometer until the same reading is obtained on the feeler gage. This method can give results to within 0.05 pct elongation at all strains.

† X-ray residual strains were recorded below the 0.2 pct yield strength, but not below the elastic limit (within the limits of experimental error).

The back-reflection X-ray technique was employed.‡ Three exposures at each stress were taken; one perpendicular to the axis of the specimen and two oblique at an angle of 45° to the axis. Tungsten powder was used as a reference material. A thin film of the powder was placed on the specimen using petrolatum as the adhesive. For the low temperature tests, a thin plastic tube was placed closely around the outside of the specimen, supported on the lower collar. The reference material was placed on this to assure that the solidified powder-petrolatum mixture would not undergo any straining due to the strain applied to the specimen itself. Cobalt radiation was used. The (420) planes of the 61S aluminum alloy and the (222) planes of the tungsten reference powder were the reflecting planes.

‡ For details see, e.g., ref. 4.

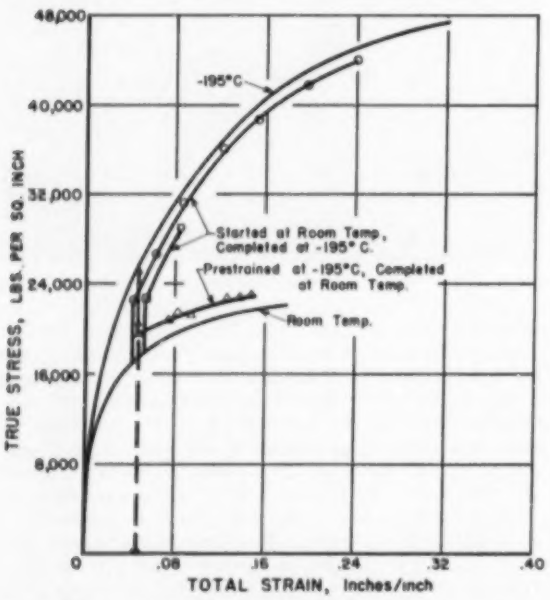


Fig. 4—True stress-strain curves for 61S-0 aluminum at room temperature and at —195°C and with crossover for testing temperatures.

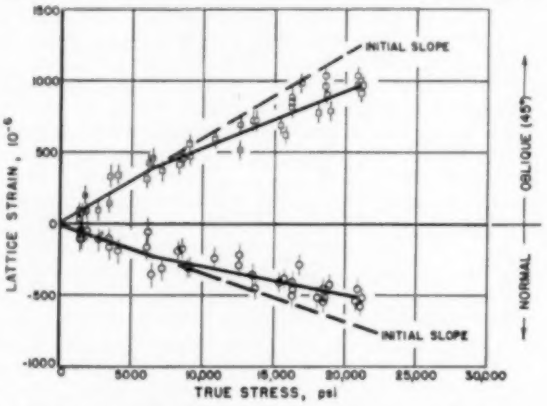


Fig. 5—Lattice strain-true stress curves for room temperature.

The depth of penetration of the X-rays for 90 pct absorption was 0.002 in., which was approximately 1 to 3 grains deep. Surface phenomena will, therefore, have an influence upon the results obtained. Measurement of a film yielded strain readings with a probable error of approximately ± 20 microin. per in. for stresses below the elastic limit. This probable error increases progressively to ± 60 microin. per in. at stresses corresponding to plastic deformations up to 13 pct at room temperature and 8 pct at low temperature due to the consequent X-ray line broadening. Beyond these strains the line broadening becomes too great for any reliable measurements. Reproducibility of measurements on identical pictures (for example, on the two oblique pictures taken at the same spot) was within these limits. Fig. 3 shows four typical X-ray photograms.

Results

Standard true stress-strain curves for the particular specimens used are shown in Fig. 4. The precision of the stress measurements was at worst ± 500 lb per sq in. Four sets of curves are shown; one for room temperature tests, one for tests at —195°C, one for specimens started at room temperature and

finished at −195°C, and one for specimens started at −195°C and completed at room temperature. The room temperature values for yield strength, ultimate strength, and elongation agree well with available data. The values of yield strength determined by the 0.2 pct offset method were 7700 psi at room temperature and 9700 psi at −195°C. It can be seen that at −195°C the ultimate strength and elongation at fracture have increased appreciably, while the yield strength has increased only slightly. The crossover temperature curves confirm previous findings.[1] The specimens started at room temperature then tested at −195°C do not quite reach the stress-strain curve for specimens tested entirely at −195°C. Specimens started at −195°C then changed to room temperature go higher than the room temperature stress-strain curves.

The lattice strain-true stress results at room temperature and at −195°C are summarized in Figs. 5 and 6, respectively, with the true stress as the independent variable. Each curve presents data from at least six specimens. The best fit lines are shown in each case. The scatter of points is greater than the reproducibility of measurements. This is due probably to the fact that a different sample of grains is inspected on each picture, even on the same specimen. The lattice parameter at −195°C

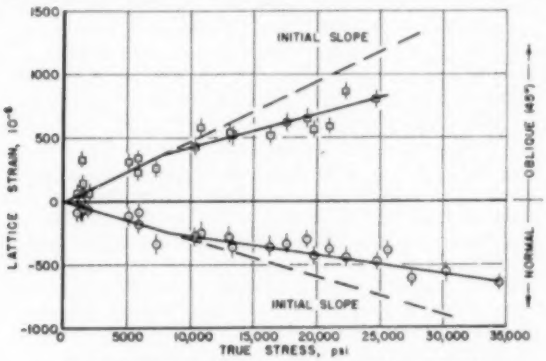


Fig. 6—Lattice strain-true stress curves for −195°C.

has, of course, decreased. This has been taken into account in calculating the strain. The observed lattice parameter at −195°C checks closely with the value calculated from the known room temperature value and the known thermal expansion data. Up to the elastic limit the curves are straight lines passing through the origin. Beyond the elastic limit, in each case, the lines depart from the initial slope in the "soft" direction. That is, the rate of increase of lattice strain with the applied stress is smaller than below the elastic limit. At a given amount of stress above the elastic limit, the low temperature results show a slightly greater deviation from proportionality than the room temperature tests. Upon unloading, from a stress above the elastic limit there is a residual strain left, Figs. 7 and 8. The greater the deviation from the original slope, the greater the residual strain. It can be noted that the rate of increase in residual lattice strain at −195°C is slightly greater than at room temperature. The initial slope of the curves for room temperature indicates a modulus of elasticity about 10 pct smaller than the average modulus of elasticity, while at −195°C the initial slopes indicate a modulus of elasticity 4½ pct smaller than the average.§

§ These moduli have been computed under assumption of isotropy. See ref. 2.

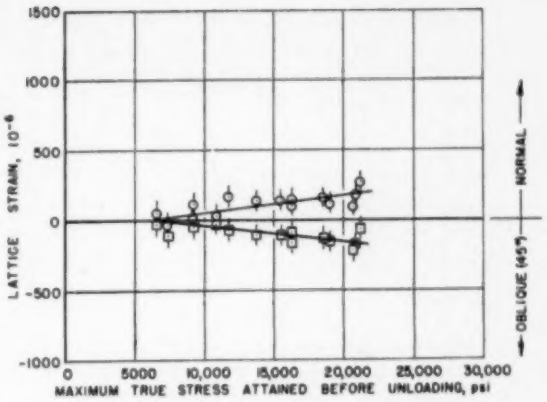


Fig. 7—Residual lattice strains at room temperature.

Absence of recovery and phase change effects was shown by measuring the residual strain on the same specimen over an interval of one month. No appreciable differences were found.

The crossover temperature results are shown in Figs. 9 and 10. Fig. 9 shows the results of a test begun at −195°C, followed by complete unloading from 24,000 lb per sq in. and reloading at room temperature. No measurements were made during unloading. The stress from which the specimen was unloaded at −195°C was close to the maximum stress attainable at room temperature; therefore, it is difficult to discern whether the reloading line actually joins the room temperature curve or passes above it. The advisability of repeating this test at a lower stress level is discussed subsequently. The crossover in the opposite direction supplied better results, Fig. 10, although more specimens would be desirable. No unloading was done here. The specimens were started at room temperature, and with the load held constant, were cooled to −195°C and the test continued. The lattice strain in these specimens did not join the −195°C curve, but appeared to fall somewhere between the room temperature and −195°C curves.

Discussion of the Results

Lattice Strain-True Stress Relationship: Figs. 5 and 6 indicate that the lattice strains are proportional to the applied stress below the macroscopically observed elastic limit but that they increase less than proportionally beyond this value. This finding is in agreement with the results reported previously for 61S aluminum alloy[2] and other

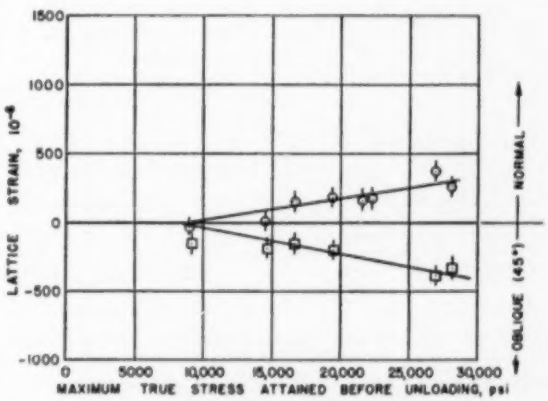


Fig. 8—Residual lattice strains at −195°C.

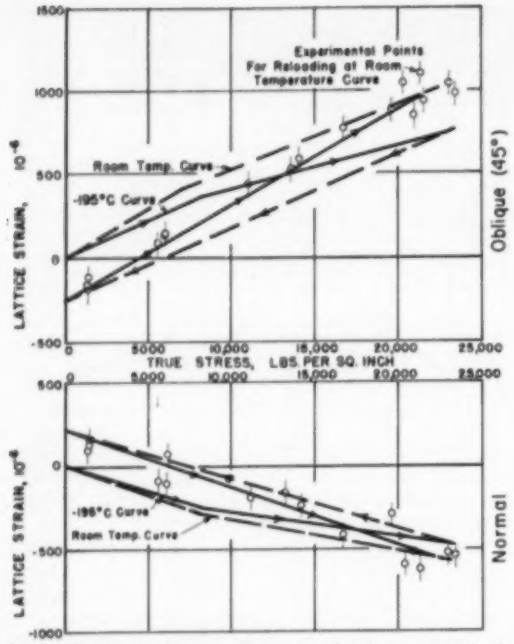


Fig. 9—Lattice strain-true stress cycle. Specimens prestrained at —195°C, test completed at room temperature.

alloys[7] at room temperature. The deviation from proportionality has been variously interpreted. However, the general consensus at present appears to be that the major contributing factor is some sort of structural heterogeneity which causes various parts of the crystalline aggregate to flow at different yield stresses.[8] According to this interpretation, the deviation from proportionality observed in Figs. 5 and 6 is due to the fact that the grains contributing to the (420) diffraction pattern in the normal and oblique (45°) exposures are not randomly distributed among all parts of the aggregate, but represent a sampling of only some of the parts. A greater deviation from proportionality is observed in the low temperature tests. This would indicate that the sample is farther removed from the average than at room temperature. Since the number of grains contributing to the X-ray pattern is the same in both cases, the result can be interpreted as being due to a wider spread of yield stress values at low temperature.

Controversy exists as to the nature of the heterogeneity responsible for this spread.[9] This point is beyond the scope of this paper and will not be considered here.

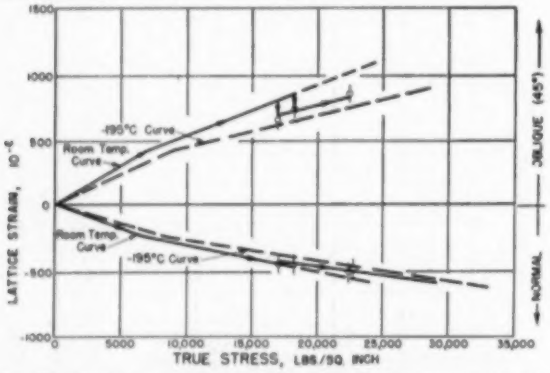


Fig. 10—Lattice strain-true stress cycle. Specimens started at room temperature, test completed at —195°C.

Residual Lattice Strains: If upon unloading from a stress beyond the elastic limit, a straight line were followed parallel to the slope at the origin, Figs. 5 and 6, a residual lattice strain would be found equal to the intercept made on the vertical axis. For a "soft" orientation the residual strain is of an opposite sign to the strain measured under load. This conclusion has been verified experimentally as to the sign. However, on the basis of the best fit, the measured residual strains are smaller for the oblique exposures, Figs. 7 and 8, indicating a possible deviation from the straight line during unloading. This phenomenon would be analogous to that observed with the macroscopically measured strain[4] and it could be similarly explained as resulting from the Bauschinger effect. On the other hand, the process of reloading seems to follow a straight line parallel to the slope at the origin within the limits of experimental error, Fig. 9.

Equation of State: Inspection of Figs. 4 and 10 shows a definite similarity of behavior between the lattice strain, especially in the oblique exposure, and the macroscopically measured strain. Here, as there, the specimens started at room temperature, then changed to —195°C, do not quite reach the stress-strain curve for specimens tested entirely at —195°C. This divergence shows dependence on previous history and failure of the equation of state. It is of interest to note that the crossover points in the oblique exposure which lie above the stress-strain curve at —195°C can be obtained graphically by following the same type of path as shown in Fig. 9. That is, the same crossover points are obtained if the specimen is assumed to have been unloaded at room temperature, to have acquired a residual strain given by the diagram, Fig. 7, and to have followed a straight line parallel to the slope at the origin at —195°C on reloading. If the same construction is applied in reverse order starting from the actual curve at —195°C and using the corresponding data of Fig. 8, it will be found that the crossover points are also slightly above the room temperature stress-strain diagram, but that the difference is well within the experimental error. This finding would seem to indicate that there would be little advantage in repeating the tests of Fig. 9 at lower stress levels. Likewise, the curves for the normal exposures at room temperature and at —195°C are too little apart to afford a sensitive criterion of the equation of state. Therefore, the failure of the equation of state for the lattice strains appears to rest solely on the evidence supplied by the oblique stress-strain curves of Fig. 10.

Conclusion

On the basis of the results obtained with the 61S aluminum alloy it does not appear that X-ray or lattice strains constitute a suitable parameter for an equation of state. However, additional tests on other materials must be performed before a more definite statement can be made.

References

[1] J. E. Dorn, A. Goldberg, and T. E. Tietz: The Effects of Thermal-Mechanical History on the Strain Hardening of Metals. *Trans.* AIME (1949) **180**, p. 205; METALS TECHNOLOGY (September 1948).

[8] G. B. Greenough: *Progress in Metal Physics* (1952) **3**, p. 176.

[9] J. P. Frankel: Ph.D. Thesis, Dept. of Engineering, Univ. of California, Los Angeles, 1951.

[4] C. S. Barrett: *Structure of Metals* (1952) New York. McGraw-Hill Book Co.

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aime NEWS

Iron and Steel Makers Set "Meeting of the Year"

Blast Furnace and Open Hearth Sessions Cover All Operating and Technical Developments

Meeting to be at the Palmer House, Chicago, April 5 to 7, 1954

THE 37th Conference of the Blast Furnace, Coke Oven, and Raw Materials Committee and National Open Hearth Steel Committee will be held at the Palmer House, Chicago, Ill., Apr. 5 to 7, 1954. All operating men, repair men, metallurgists, ceramic and refractory engineers, observers, and any others interested in the production of more steel, better steel, and cheaper steel and in any of the problems connected with the reduction of iron ore in the blast furnace, are invited to attend.

The registration fee for either Conference is $7, which includes a copy of the respective Proceedings when printed. Students pay no registration fee, unless they wish a copy of the Proceedings. AIME Members are entitled to a $2 reduction in the registration fee upon presentation of their membership card.

Hotel reservation requests should be sent directly to the hotel of your choice as soon as possible. Mention the Blast Furnace or Open Hearth Conference of AIME, as well as time of arrival. Hotels will not hold blocks of rooms assigned after March 15.

Simultaneous inspection trips have been planned for Wednesday, April 7, through the courtesy of Inland Steel Co. and the Continental Foundry & Machine Co. The Continental plant will be of interest to those interested in cold metal operations and/or acid open hearth practice. Chartered buses will leave the Palmer House at 9:00 am. Luncheon arrangements have been taken care of by the host companies. A charge of $2.50 will be made by the Committees to defray plant trip transportation costs.

Preliminary Program of Blast Furnace, Coke Oven, and Raw Materials Conference

SUNDAY, APRIL 4

1 to 9 pm
Registration

MONDAY, APRIL 5

8 am to 4 pm
Registration

10:00 am
General Session:
Welcoming remarks. Fire clay for blast furnaces; burdening blast furnaces with various iron bearing materials.

2:00 pm
Agglomeration and Raw Materials Session:
Sintering conditions and sinter properties as effected by moisture and fuel; effect of additives on properties of sinter; sinter is what you make it.

TUESDAY, APRIL 6

8 am to 4 pm
Registration

9:30 am
Blast Furnace Gas Session:
Ferromanganese gas cleaning installation; continuous top gas analysis; observation on blast heat.

9:30 am
Coal and Coke Session:
Use of washed coal for coke; effect of hot water quenching on coke; measuring expansion pressure during carbonization.

12:30 pm
Luncheon Meeting:
Everyone attending the Blast Furnace, Coke Oven and Raw Materials sessions is invited to attend this luncheon and annual business meeting.

2:00 pm

Blast Furnace Theory Session:
Iron ore reduction; slag metal reactions; flow of gases and heat transfer through packed solids.

6:30 pm

Reception

7:00 pm

Annual Fellowship Dinner
Speaker: John T. Rettaliata, president, Illinois Institute of Technology

WEDNESDAY, APRIL 7

8 am to 4 pm

Registration

9:00 am

Plant trip

Preliminary Program of National Open Hearth Committee

SUNDAY, APRIL 4

1 to 9 pm

Registration

MONDAY, APRIL 5

8 am to 4 pm

Registration

10:00 am

General Session
Welcoming remarks. Announcements; McKune Award paper.

10:30 am

Hot Metal Basic Operations Session:
Scrap quality; tapping heats into two ladles; cleaning waste gases; aids to labor.

10:30 am

Acid Operating Session:
Rammed bottoms; maintenance and care of bottoms; pouring of large ingots; resume of sulphur investigation.

2:00 pm

Refractories and Masonry Session:
Hot repair or new roof; strength of basic brick at high temperatures; control testing of refractories; checker cleaning methods.

2:00 pm

Role of Manganese in Steelmaking Session:
Mn modification of Fe-O-S system; Mn supply; oxidation of P and Mn during and after flushing; behavior of Mn.

TUESDAY, APRIL 6

8 am to 4 pm

Registration

9:30 am

Operating Metallurgy Session:
European steelmaking practice; pouring conditions as revealed by high speed motion pictures; effect of rare earth additions on surface quality; factors affecting performance.

2:00 pm

Shop Operating for Maximum Production, Furnace Life and Quality Session:
Establishing the pattern; stocking and charging; furnace equipment and instrumentations: control of melt carbon and working heat; bottom making.

2:00 pm

Cold Metal and Basic Foundry Session:
Elimination and control of air infiltration; steel quality with low iron charges; charging and fluxing with low pig iron and burnt lime; restricted uptake furnaces; furnace design for 100 pct natural gas firing.

6:30 pm

Reception

7:00 pm

Annual Fellowship Dinner
Speaker: John T. Rettaliata, president, Illinois Institute of Technology

WEDNESDAY, APRIL 7

8 am to 4 pm

Registration

9:00 am

Plant trip

Hartford, Conn., Is Scene Of New England Conference

THE Eighth Annual New England Regional Conference of the Institute of Metals Div., AIME will be held at the Hotel Bond, Hartford, Conn., April 30 to May 1, 1954.

Registration will be in the main lobby of the Hotel Bond. Fee for the registration and banquet will be $6.00 and $2.00 for registration only. There will be no charge for student registration. The speaker for the banquet will be Glen B. Eastburn. Reservations should be made, with remittance, before Apr. 20, 1954 to Leo P. Tarasov, research & development dept., Norton Co., Worcester, Mass. Make checks payable to Mr. Tarasov.

Requests for hotel reservations should be sent directly to the hotel, mentioning the Conference.

The plant tour will be to the Pratt & Whitney aircraft div., United Aircraft Corp., East Hartford, Conn. Bus transportation will be available from the Hotel Bond. The tour of the plant will cover manufacture

of various engine parts, both resistance and fusion welding, brazing and heat treatment of engine components, testing of metals and alloys and witnessing an aircraft engine under test. Persons registering for the plant visitation should note if they are U. S. citizens. Noncitizens will have to get special clearance.

Chairman of the Conference Committee is Winston H. Sharp. Serving with him on the Committee are: Franklin H. Wilson, American Brass Co., Vice-Chairman; William L. Stearns, Bridgeport Brass Co., Secretary; Leo P. Tarasov, Norton Co., Treasurer; James H. Moore, Vacuum Metals Corp., Past Chairman; Walter A. Backofen, Massachusetts Institute of Technology; Gerald R. Brophy, International Nickel Co.; Richard J. Christine, American Brass Co.; Martin B. Graham, Boston Naval Shipyard; William D. Robertson, Yale University; and Richard E. Russell, Chase Brass & Copper Co.

FRIDAY, APRIL 30

9:00 am

Registration

9:30 am

Plant Visit

2:00 pm

Metals Engineering

G. R. Brophy, Chairman

Large Light Metal Forging: A. J. Pepin, Wyman-Gordon Co., Worcester, Mass.

Powder Metallurgy: Claus G. Goetzel, Sintercast Corp. of America, Yonkers, N. Y.

Recent Developments in Rolling Metals: N. H. Polakowski, Armzen Corp., Waterbury, Conn.

Modern Casting Practices: F. B. Herlihey, American Brake Shoe Co., Mahwah, N. J.

6:00 pm

Cocktail Hour

7:00 pm

Banquet

Helicopter Operations in Metropolitan Areas: Glen B. Eastburn, New York Airways, Inc.

SATURDAY, MAY 1

9:30 am

Transformations in Alloys

W. A. Backofen, Chairman

Fundamentals of Phase Transformations: Morris Cohen, Massachusetts Institute of Technology, Cambridge, Mass.

Phase Transformations in Titanium Alloys: Leonard S. Jaffee, Watertown Arsenal, Watertown, Mass.

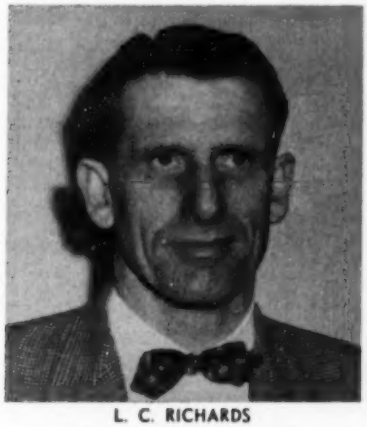
Phase Transformations in Copper Alloys: E. W. Palmer, F. H. Wilson, American Brass Co., Waterbury, Conn.

12:30 pm

Luncheon

Program Set for Divisions Participating in Northwest Conference

F. X. CAPPA
General Chairman



L. C. RICHARDS
Ind. Min. Div. Chairman

Francis X. Cappa, smelting div. mechanical engineer at the Vancouver, Wash., works of the Aluminum Co. of America, is serving as General Chairman of the AIME Pacific Northwest Metals and Minerals Conference to be held Apr. 29 to May 1, 1954, at the Multnomah Hotel in Portland, Ore.

The Metals Branch program is in charge of the following division chairman: ISD, G. R. Heffernan, general superintendent, Vancouver Rolling Mill, Ltd.; EMD, H. A. Lee, superintendent of metallurgy and research, Bunker Hill & Sullivan Mining Co., Kellogg, Idaho; IMD, S. E. Maddigan, div. of metallurgical research, Kaiser Aluminum & Chemical Co., Spokane.

Papers to be presented in the Iron and Steel Div. will cover iron ores, coal and coke, power and fuel, and processes as related to the Pacific Northwest. EMD will highlight trends in extractive metallurgy, uranium ores, tungsten, indium, and include a symposium on synthetic materials for filtering dust and fume.

The physical metallurgy program will include papers on titanium alloys, aluminum, tin and antimony, weldability of ductile iron, effects of ultrasonics on carburizing, high pressure oxidation, and dislocation boundaries. The Metals Branch luncheon speaker will be Dr. William A. Pearl, Administrator, Bonneville Power Administration.

Leslie C. Richards, mining engineer of Portland, Ore., is the Pacific Northwestern Chairman for the Industrial Minerals Div. Ind. Min. Div. participation in the Pacific Conference will consist of three sessions. One of these will feature a symposium on ground water resources with A. M. Piper, staff scientist of the USGS, as moderator.

Two other sessions will be devoted to nine papers on the occurrence, mining, processing, transportation, and marketing of the West's nonmetallics. Chairmen for these are: T. M. Robins, Major General U. S. Army, Ret., president, Raw Materials Survey, Inc. Portland, Ore.; V. E. Scheid, dean, Mackay School of Mines, University of Nevada; H. W. Marsh, secretary, Idaho Mining Assn.; and G. H. Waterman, president, Manufactures Minerals Co., Seattle.

An Industrial Minerals Div. luncheon is being planned which will feature as speaker the 1954 National Chairman of the Div., Robert C. Stephenson.

There will also be one technical session on applied geology and an evening session on mineral industry education. The Grand Banquet will be Friday, April 30. The JOURNAL OF METALS will publish the complete program for the Metals Branch in the April issue.

William E. Wrather Honored at Waldorf-Astoria Dinner

William E. Wrather, director of the U. S. Geological Survey, Washington, D. C., received the John Fritz Medal at a dinner in his honor at the Waldorf-Astoria, New York. The medal is given jointly by the four founder societies, American Institute of Mining and Metallurgical Engineers, of which Mr. Wrather is a Member; American Society of Civil Engineers; American Society of Mechanical Engineers; and American Institute of Electrical Engineers. Seated from left to right, outer rim: John Keshishian, T. B. Counselman, Erle V. Daveler, Louis S. Cates, John M. Lovejoy, Felix E. Wormser, Fred M. Nelson, John Suman, William E. Wrather, Andrew Fletcher, D. H. McLaughlin, Edward H. Robie, J. Ed. Warren, J. Terry Duce, Michael L. Haider, James Boyd, Reginald Burbank, and O. B. J. Fraser. Seated on the inside rim, from left to right, are: Willis Mc. G. Peirce, Russell B. Caples, A. B. Kinzel, C. M. Cooley, Clyde Williams, E. O. Kirkendall, H. DeWitt Smith, Thomas B. Nolan, Ernest Hartford, M. D. Cooper, and James L. Head.

Australia Is Topic At Spokane Meeting

The January 15 meeting of the Columbia Section AIME in Spokane was highlighted by S. L. Channon's talk on the mineral industry of Australia. Dr. Channon, a native of Australia, described the vicissitudes of the mining industry from the 1870's to the present time. A movie *Advance Australia* augmented the talk and gave a good overall view of life and activity in that country. Dr. Channon is a metallurgist, dept. of metallurgical research of Kaiser Aluminum & Chemical Corp.

The meeting scheduled for March 19 at Spokane will have as its topic *Mining and Metallurgy at Pend Oreille*. John Fretts, geologist, L. G. Gillings, mine superintendent, and J. C. Crampton, mill superintendent of the Pend Oreille Mining Co. will discuss the subject.

Bridgeport Brass Co. Host at AIME Meeting

The third technical session of the Connecticut Section AIME was held January 13 at the Bridgeport Brass Co., Bridgeport, Conn. Arthur Young, spectrographer of the Bridgeport Brass Co., spoke on the direct-reading spectrometer. Mr. Young traced the development of this type of equipment through its initial use for magnesium alloys in 1945, aluminum alloys in 1946, ferrous alloys in 1947, and copper alloys in 1951. All elements except arsenic, phosphorus, and antimony can be analyzed spectrographically in quantities up to 42 pct with one instrument setting.

Following this presentation a discussion panel composed of Mr. Young, E. Wade, Scoville Mfg. Co.; R. Ray, American Brass Co.; and P. Leichtle, Chase Brass & Copper Co., offered comments on the subject.

The tentative plans for the meeting to be held March 10 at the Torrington branch of the American Brass Co. include a plant visit in the afternoon and a technical session entitled, *Brass Mill Seminar*.

New York Group Meets At Columbia

On January 19 the Physical Metallurgy Group of the New York Section AIME held their second meeting of the year 1953-54 at Columbia University. The program started with a tour through the metallurgical laboratories of the School of Mines. After dinner R. D. Heidenreich of the Bell Telephone Laboratories presented a talk on *Electron Metallography of Precipitation and Phase Transformations*.

Mr. Heidenreich discussed the transition structure in lead-silver alloys as revealed by the electron microscope, and the effect of a few hundredths of 1 pct Ag on the mobility of dislocations. Electron microscopy as used in the study of the decomposition of austenite has also demonstrated that upon repeated heating and cooling through the A_1 temperature there is a pronounced *memory* for twins in the austenite field.

Scientific Congress To Be Held in Paris

The *L'Aluminium Français* and *La Société Chimique de France* expect to hold a scientific congress and exhibition in Paris from June 14 to 19, 1954 to celebrate the centenary of the first production of industrial aluminum in France by Henry Sainte-Claire Deville.

The activities of the congress will consist of a meeting at the Sorbonne in memory of Sainte-Claire Deville on Monday, June 14; a debate on papers dealing with aluminum metallurgy, the chemistry of aluminum compounds, and the uses of aluminum and aluminum alloys on June 15 to 16; and visits to plants and laboratories in France from June 17 to 19. In addition there will be an exhibition of aluminum and aluminum products prepared by *L'Aluminium Français* in Paris during the congress.

AIME members interested in obtaining final programs and other relevant information should contact: R. Gadeau, The Secretary, L'Aluminium Français, 23 Rue Balzac, Paris (8e), France.

Apr. 15, 1954 is the deadline for papers that are to be presented at the Institute of Metals Div. Fall Meeting in Chicago, Nov. 1 to 3, 1954. Papers received by this deadline but requiring revision may not be processed in time to permit scheduling for this meeting.

Brazil Scene Of 1954 Summer Meetings

As part of the year long observance of the 400th Anniversary of the founding of the city of Sao Paulo, a large number of international organizations are holding meetings and conventions in Brazil. Three of these meetings which are of particular interest and importance to engineers have been scheduled for this summer.

The Sectional Meeting of the World Power Conference will take place in Rio de Janeiro, July 25 to August 10. The U. S. National Committee for the World Power Conference is working with the Dept. of State in organizing the U. S. delegation. The Interamerican Assn. of Sanitary Engineers will meet from July 25 to 31, and the U. S. delegation is in the process of being formed.

The Third Convention of the Pan-American Federation of Engineering Societies (UPADI) will meet in Sao Paulo, August 2 to 12. This meeting will attract engineers of all societies from Latin America, Canada, and the United States. Engineers Joint Council is currently selecting the U. S. delegates and alternates.

Information may be secured from the North Shore Travel Center at 609 Plandome Rd., Manhassat, Long Island, N. Y. or from the sponsoring organizations.

The meeting of the AIME Bessemer Steel Committee was held at the Duquesne Club on Jan. 13, 1954. First Row: J. J. Golden, U. S. Steel Corp.; W. M. Zatezalo, Weirton Steel Co.; C. M. Kay, U. S. Steel Corp.; W. D. Smith, Jones & Laughlin Steel Corp.; J. P. Ely, National Tube div., U. S. Steel Corp.; R. E. Edwards, Jones & Laughlin Steel Corp. Second Row: P. E. Lindberg, Jr., McLouth Steel Corp.; C. D. King, U. S. Steel Corp.; F. J. McMulkin, Dominion Foundries & Steel Ltd.; A. B. Wilder, National Tube div., U. S. Steel Corp.; W. O. Philbrook, Carnegie Institute of Technology; H. C. Smith, Great Lakes Steel Corp.; G. M. Yocom, Wheeling Steel Corp. Third Row: H. B. Emerick, Jones & Laughlin Steel Corp.; G. McMorran, U. S. Steel Corp.; W. G. McDonough, National Tube div., U. S. Steel Corp.; E. B. Plott, Youngstown Sheet & Tube Co.; R. H. Frushour, Youngstown Sheet & Tube Co.; T. R. Bailes, Bethlehem Steel Co.; E. B. Hughes, Wheeling Steel Corp.; H. C. Dunkle, Republic Steel Corp.; R. J. Walter, Wheeling Steel Corp.; J. W. Schroeder, National Tube div., U. S. Steel Corp.; W. F. Copp, Wheeling Steel Corp.; W. H. Mayo, U. S. Steel Corp.; J. M. Stone, Tennessee Coal & Iron div., U. S. Steel Corp.; L. T. Sanchez, National Tube div., U. S. Steel Corp. Fourth Row: W. P. Holloway, Wheeling Steel Corp.; E. P. Best, A. M. Byers Co.; R. C. Johnston and J. F. Beale, Republic Steel Corp.; J. C. Weber, U. S. Steel Corp.; L. A. Xidis and A. C. Flanders, Wheeling Steel Corp.; W. R. McLain, U. S. Steel Corp.; K. Gordon and J. Evans, Weirton Steel Co.

A. E. White To Be Honored

Prof. A. E. White will be honored by a Symposium on the Utilization of Heat Resistant Alloys on the occasion of his retirement from the University of Michigan. The symposium will be held Mar. 11 and 12, 1954. Dr. White was born Mar. 12, 1884 at Plainville, Mass. He received an A.B. degree from Brown University in 1907 and took a year of graduate work in engineering at Harvard. In 1925 Brown University bestowed upon him the honorary degree of Doctor of Science. After varied experience in both industry and government, Professor White became director of the Engineering Research Institute of the University of Michigan. He has held this post for 34 years.

The symposium is divided into four sessions: General Principles for Utilization of Heat Resistant Alloys, Engineering Practice in Design and Selection of Materials for Heat Resistant Applications, Production and Fabrication Aspects of the Use of Heat Resistant Alloys, and Metallurgical Variables—Specification and Acceptance Testing.

Coming Events

Mar. 2-4, SAE, Hotel Statler, Detroit.

Mar. 3, AIME, Chicago Section, Chicago Bar Assn., Chicago.

Mar. 4-5, American Society for Metals, mid-winter meeting, Hotel Statler, Boston.

Mar. 8-10, American Institute of Chemical Engineers, Statler Hotel, Washington, D. C.

Mar. 10, AIME, Connecticut Section, American Brass Co., Torrington, Conn.

Mar. 10-12, ASME, international meeting, Hotel Del Prado, Mexico City.

Mar. 15-19, National Assn. of Corrosion Engineers, Municipal Auditorium, Kansas City.

Mar. 17, AIME, National Open Hearth Steel Committee, Western Section, Rodger Young Auditorium, Los Angeles.

Mar. 19, AIME, Spokane Subsection.

Mar. 24-26, American Power Conference, annual meeting, Sherman Hotel, Chicago.

Apr. 5-7, AIME, Blast Furnace, Coke Oven, Raw Materials Conference, Palmer House, Chicago.

Apr. 5-7, AIME, National Open Hearth Conference, Palmer House, Chicago.

Apr. 5-7, American Society of Lubrication Engineers, annual meeting and exhibit, Cincinnati.

Apr. 6-9, Institute of Physics, conference on physics of particle size analysis, University of Nottingham, Nottingham, England.

Apr. 7, AIME, Chicago Section, Chicago Bar Assn., Chicago.

Apr. 7-8, Inland Empire Science Fair, Lewis & Clark High School, Spokane, Wash.

Apr. 21-23, Southern Industrial Wastes Conference, Hotel Shamrock, Houston.

Apr. 24, AIME, Columbia Section, student meeting, Idaho University, Moscow, Idaho.

Apr. 26-28, Canadian Institute of Mining and Metallurgy, annual meeting, Mount Royal Hotel, Montreal.

Apr. 26-28, Metal Powder Assn., show and annual meeting, Drake Hotel, Chicago.

Apr. 26-30, American Society of Tool Engineers, industrial exposition, Convention Center, Philadelphia.

Apr. 27, Assn. of Consulting Chemists and Chemical Engineers, Hotel Belmont Plaza, New York.

Apr. 29-May 1, AIME, Pacific Northwest Metals and Minerals Conference, Multnomah Hotel, Portland, Oregon.

Apr. 30-May 1, AIME, New England Regional Meeting, Bond Hotel, Hartford, Conn.

May 2-6, Electrochemical Society, La Salle Hotel, Chicago.

May 4-7, American Welding Society, national meeting, Hotel Statler, Buffalo.

May 7-8, Pennsylvania Society of Professional Engineers, annual state convention, Bedford Springs Hotel, Bedford, Pa.

May 8-14, American Foundrymen's Society, Cleveland Auditorium, Cleveland.

May 16-17, American Institute of Chemical Engineers, Kimball Hotel, Springfield, Mass.

May 31-June 11, Canadian International Trade Fair, Toronto.

June 14-18, American Society for Engineering Education, annual meeting, University of Illinois, Urbana, Ill.

June 20-23, Chemical Institute of Canada, Toronto, Canada.

June 20-25, American Institute of Chemical Engineers, nuclear energy meeting, University of Michigan, Ann Arbor, Mich.

July 16-21, Joint Commission on Electron Microscopy, international conference, Senate House, University of London; London School of Hygiene and Tropical Medicine, Malet St., London, W. C. 1, England.

July 19-20, International Conference, Mechanical Effects of Dislocations in Crystals, University of Birmingham, England.

July 21-28, International Union of Crystallography, general assembly and international congress, Paris.

Sept. 14-24, Instrument Society of America, Philadelphia.

Sept. 20-24, American Mining Congress, Civic Auditorium, San Francisco.

Oct. 3-7, Electrochemical Society, Inc., Statler Hotel, Boston.

Oct. 26, Assn. of Consulting Chemists and Chemical Engineers, Inc., annual symposium and banquet, Hotel Belmont Plaza, New York.

Personals

J. Franklin Miller, formerly with the Freyn Engineering div., Koppers Co., Inc., Chicago has been appointed to the international dept. staff at the Armour Research Foundation of the Illinois Institute of Technology. Mr. Miller will be located in Rangoon, Burma to take part in an industrial development program being carried out by the Foundation.

Francis Thomas, vice-president of Orinoco Mining Co., has been elected president of the U. S. Steel Corp. subsidiary. He succeeds **Mack C. Lake,** who will continue with the company in a consulting capacity.

NEVILLE S. SPENCE

Neville S. Spence formerly with Dominion Magnesium Ltd., Haley, Ontario, has accepted a position with the Canadian Dept. of Mines & Technical Surveys, Chalk River, Ontario.

Hugh T. Smith is with the Linde Air Products Co., St. Louis, Mo.

George Shutak has been appointed to the sales engineering staff of the Cleveland-Youngstown office of the Ramtite Co.

W. J. Ulrich, senior sales representative, Detroit, Carborundum Co., was elected vice-president of the company.

Robert L. Larson was appointed manager of the alloy steel div., Joseph T. Ryerson & Son, Inc. He was formerly manager of alloy steel sales, Chicago. **William P. Loehrer** succeeds Mr. Larson.

W. A. Anderson was recently appointed manager of operations, Pittsburgh Screw & Bolt Corp., for the Pittsburgh and Graham plants. He had been assistant manager of operations since 1952.

James E. Reynolds is with Battelle Memorial Institute, Columbus, Ohio.

Edgar B. Speer, formerly steelmaking superintendent, div. superintendent steel products, U. S. Steel Co., Gary, Ind., has been transferred to Duquesne, Pa., as assistant general superintendent.

David B. Scott has joined the Revere Copper & Brass Co., Chicago, as a metallurgist. He was formerly with the American Can Co., Maywood, Ill.

Gordon V. Richdale has been elected as president of Engelhard Industries Group. **Charles W. Engelhard** will continue as chairman and general manager.

Edmond Charles Falleur was recently appointed chief technical engineer for the Belgian Electric Sales Corp., New York.

Latham E. Osborne was appointed executive vice-president, Westinghouse Electric Corp. He was formerly executive vice-president, defense products. He was also elected to the board of directors.

Robert S. Beckham was named vice-president in charge of distribution, Multi-Amp Corp., Harrison, N. J.

I. R. Warshow, assistant div. controller, Doehler-Jarvis div., National Lead Co., has been named controller.

L. Douglas Welford has been named assistant to the executive vice-president in charge of Government relations, Stauffer Chemical Co., Washington, D. C.

Emil A. Johnson and **A. C. Barackman,** have retired from the Wheeling Corrugating Co., Wheeling, W. Va. Mr. Johnson had been with the company 44 years and was manager of the Minneapolis branch. Mr. Barackman joined the company 42 years ago and was manager of the Detroit branch.

J. M. Weldon has been transferred to the general sales dept., International Nickel Co., Inc., as assistant to the vice-president.

Donald J. Leehey, was recently appointed manager of the Atomic Energy Commission operations at Santa Fe.

J. H. Lindemuth, Kaiser Aluminum & Chemical Corp., has been transferred from Chalmette, La., to Oakland, Calif.

Kent M. Campbell has been named manager of Solar Aircraft Co.'s Dallas-Fort Worth branch office.

STANLEY F. REITER

Stanley F. Reiter has resigned from the General Electric Co., research laboratory and is now associated with the Rome Fastener Corp., New Haven, Conn., as technical director.

G. H. Hille has been appointed director of purchases for Salem-Brosius Inc., Salem, Ohio.

D. H. W. Felch has been appointed Far Eastern representative by the American Cyanamid Co., and will be located in Tokyo.

J. D. Kennedy, manager of product projects at the Carboloy dept., General Electric Co., Detroit, has been named manager of the dept.'s newly created South Central sales district with headquarters in St. Louis.

John Brandesburg, sales manager for Amsco div., has been appointed vice-president in charge of sales, American Brake Shoe Co., Amsco div.

John H. Horlick, Jr., manager of the technical service div., explosives dept., Hercules Powder Co., has retired after 39 years with the company. **Milo A. Nice** has been appointed to succeed Mr. Horlick.

Herbert A. Long, former chief engineer, Pittsburgh Steel Co., Allenport, Pa., has been named chief engineer for the company. **Donald C. Duvall** was appointed works manager at the Monessen plant, succeeding **William J. Huston,** who is retiring. **Clarence M. Feehan** has been promoted from assistant superintendent of the open hearth dept., Monessen to superintendent, succeeding **Karl Leinberger,** who is retiring.

W. M. Trigg has been named manager of Westinghouse Electric Corp.'s new metals development plant which will be built at Blairsville, Pa.

S. H. Smith, district manager of the Milwaukee office for Air Reduction Sales Co., has retired. **G. J. Dekker,** formerly district manager of St. Louis succeeds Mr. Smith as district manager of Milwaukee.

A. MacKenzie has been transferred as manager of Cleveland welds works and Jefferson welds works, lamp div., General Electric Co., and will be located in Cleveland. He had formerly been coordinator of production and personnel, parts mfg., East Cleveland for the firm.

J. N. Langley will be located in Uganda, British East Africa as metallurgical engineer for Kilembe Mines Ltd.

Sidney Rolle has retired from the Scomet Co. and the American Metal Co., Ltd., and is located in New York City as a consulting metallurgical engineer.

Charles D. Michaelson was appointed vice-president of Braden Copper Co., a subsidiary of Kennecott Copper Corp. He will be located in Santiago, Chile.

G. T. Harley, who has been manager of the New Mexico operations, International Minerals & Chemical Corp.'s potash div., has been appointed special consultant to the potash div.

Thorndike Saville, dean of New York University's College of Engineering, has been elected president of Engineers' Joint Council.

Robert L. Fischer, Boeing Aircraft Co., manufacturing research dept., has been transferred from Bettendorf, Iowa, to Seattle, Wash.

William H. Congleton, technical director of American Research & Development Corp., was elected a director of Baird Associates, Inc.

B. M. Downey, manager of the Houston plant, Shell Chemical Corp., has been appointed manager of manufacturing and **A. W. Fleer,** manufacturing operations manager, has been named manager of research, development, and engineering.

Kempton H. Roll, Lead Industries Assn., New York, has been elected chairman of the North East region, National Assn. of Corrosion Engineers, Houston, Texas.

Horace W. Higgs has been named assistant general superintendent of smelting and refining for Cerro de Pasco Corp., La Oroya. He had been superintendent of lead smelting.

R. W. Sandelin has been appointed technical director, Alloy Metal Wire Co., div., H. K. Porter Co., Inc., Prospect Park, Pa. Dr. Sandelin had been with the company's Connors steel div. as chief metallurgist.

J. J. Kraus was recently promoted to vice-president of sales for Sharon Steel Corp., Sharon, Pa. **D. A. Nabb** was named general manager of the Detroit tube & steel div.

Obituaries

Ralph Glover Mickle, Jr. (Member 1949) died on Nov. 24, 1953. Mr. Mickle was a native of Crete, Neb. He was a graduate of the South Dakota School of Mines, 1944. After graduation he joined the St. Joseph Lead Co. at Herculaneum, Mo., working on feed preparation and sintering development work.

Richard Schoof, Jr. (Member 1952) died on Dec. 4, 1953. Mr. Schoof was born at Lincoln, Ill. in 1903. From 1922 to 1926 he was employed by the Old Ben Coal Corp. In 1926 he joined the Navy for four years. At one time he was associated with the American Smelting & Refining Co. In 1940 he was again in the Navy as commissaryman chief. At the time of his death he was located at San Francisco.

William H. Nikola (Member 1940), a resident of Media, Pa., died on Dec. 17, 1953. He was a graduate of the Colorado School of Mines in 1941. Following his graduation he became an aircraft maintenance officer in the U. S. Army. In 1946 he was located at the Naval Air Material Center, Philadelphia.

Necrology

Date Elected	Name	Date of Death
1938	Harlan H. Bradt	Apr. 21, 1953
1901	Donald C. Brown	Jan. 19, 1954
1949	Charles O. Burgess	Jan. 13, 1954
1945	Dana D. Champion	Nov. 7, 1953
1938	Walter R. Dewees	Apr. 2, 1953
1947	Albert E. Gamble	March 1953
1941	Walter I. Garms	Dec. 30, 1953
1930	Reg Halliday	Dec. 31, 1953
1943	Louis S. Heilig	Jan. 28, 1954
1937	Raymond C. Kasper	Dec. 20, 1953
1936	Foreman M. Lebold	Nov. 11, 1953
1944	Leonard J. Neuman	Dec. 18, 1953
1953	Joseph A. Schlickau	Nov. 9, 1953
1948	Russell Henry Wilmot	Nov. 16, 1953

Proposed for Membership

— Metals Branch AIME —

Total AIME membership on Dec. 31, 1953 was 19,718; in addition 2195 Student Associates were enrolled.

ADMISSIONS COMMITTEE

O. B. J. Fraser, Chairman; Philip D. Wilson, Vice-Chairman; F. A. Ayer, A. C. Brinker, R. H. Dickson, Max Gensamer, Ivan A. Given, Fred W. Hanson, T. D. Jones, G. W. Lutjen, E. A. Prentis, Sidney Rolle, J. T. Sherman, F. T. Sisco, R. L. Ziegfeld.

The Institute desires to extend its privileges to every person to whom it can be of service, but does not desire as members persons who are unqualified. Institute members are urged to review this list as soon as possible and immediately to inform the Secretary's office if names of people are found who are known to be unqualified for AIME membership.

In the following list C/S means change of status; R, reinstatement; M, Member; J, Junior Member; A, Associate Member; S, Student Associate.

Alabama
Birmingham—Minter, James W. (M)
Gorgas—Stanton, Morgan T. (J)

Arizona
Inspiration—Allen, Henry, Jr. (M)

California
Castro Valley—Morton, Raymond L. (M) (C/S—A-M)
China Lake—Metzger, Guinn E. (M) (C/S—J-M)
Mill Valley—Sunday, Elmer G., Jr. (M)
San Francisco—Dunn, William G. (M)

Colorado
Leadville—Enochs, Richard C. (M) (C/S—A-M)

Connecticut
Bristol—Johnson, William R. (M) (R. C/S—S-M)

Delaware
Wilmington—Ransom, John T. (M) (C/S—A-M)

Illinois
Chicago—Colner, William H. (A) (C/S—J-A)
Chicago—Henderson, Melville B. (A)
Collinsville—Moser, Clarence (J)
East St. Louis—Geist, Norman S. (M) (C/S—A-M)
Park Forest—Feurer, William E. (A)

Indiana
Highland—Elliott, John F. (M) (C/S—A-M)
Highland—Nelson, Clarence (J)
Indianapolis—Haythorne, Percy A. (M) (R. M)

Massachusetts
Needham—McCarthy, John V. (A) (R. C/S—S-A)

Michigan
Ann Arbor—Ragone, David V. (A)

New Jersey
Paramus—Von der Porten, Kenneth G. (A)
Roselle Park—Brytczuk, Walter L. (M)
Scotch Plains—McDowell, David W. (J) (R. C/S—J-M)

New Mexico
Carlsbad—Dusell, Harry N. (J)

New York
Buffalo—Brice, William A. (A)
Halesite, L. I.—Pinto, Norman P. (M) (C/S—S-A)
Kenmore—Jack, George L. (A)
New York—Vom Baur, Carl H. (M) (R. M)

Ohio
Chagrin Falls—Mapes, Edwin J., Jr. (A)
Columbus—Schwope, Arthur D. (M) (C/S—A-M)
Steubenville—Gordon, LeRoy E. (M)
Toledo—Heer, William G. N. (M)

Pennsylvania
Bala-Cynwyd—Muldawer, Leonard (M) (C/S—A-M)
Bethlehem—Horvath, Paul J. (M)
Bridgeville—Smith, Edward S. (M)
New Kensington—Dowd, James D. (M) (C/S—J-M)
Philadelphia—Green, John R. (A)
Philadelphia—Plum, Russell V. (J)
Rochester—Lund, Robert E. (M) (C/S—A-M)

Tennessee
Oak Ridge—Betterton, Jesse O., Jr. (M) (C/S—A-M)

Virginia
Richmond—Samsonetti, S. John (M)

Washington
Pullman—Beeston, Joseph M. (A)

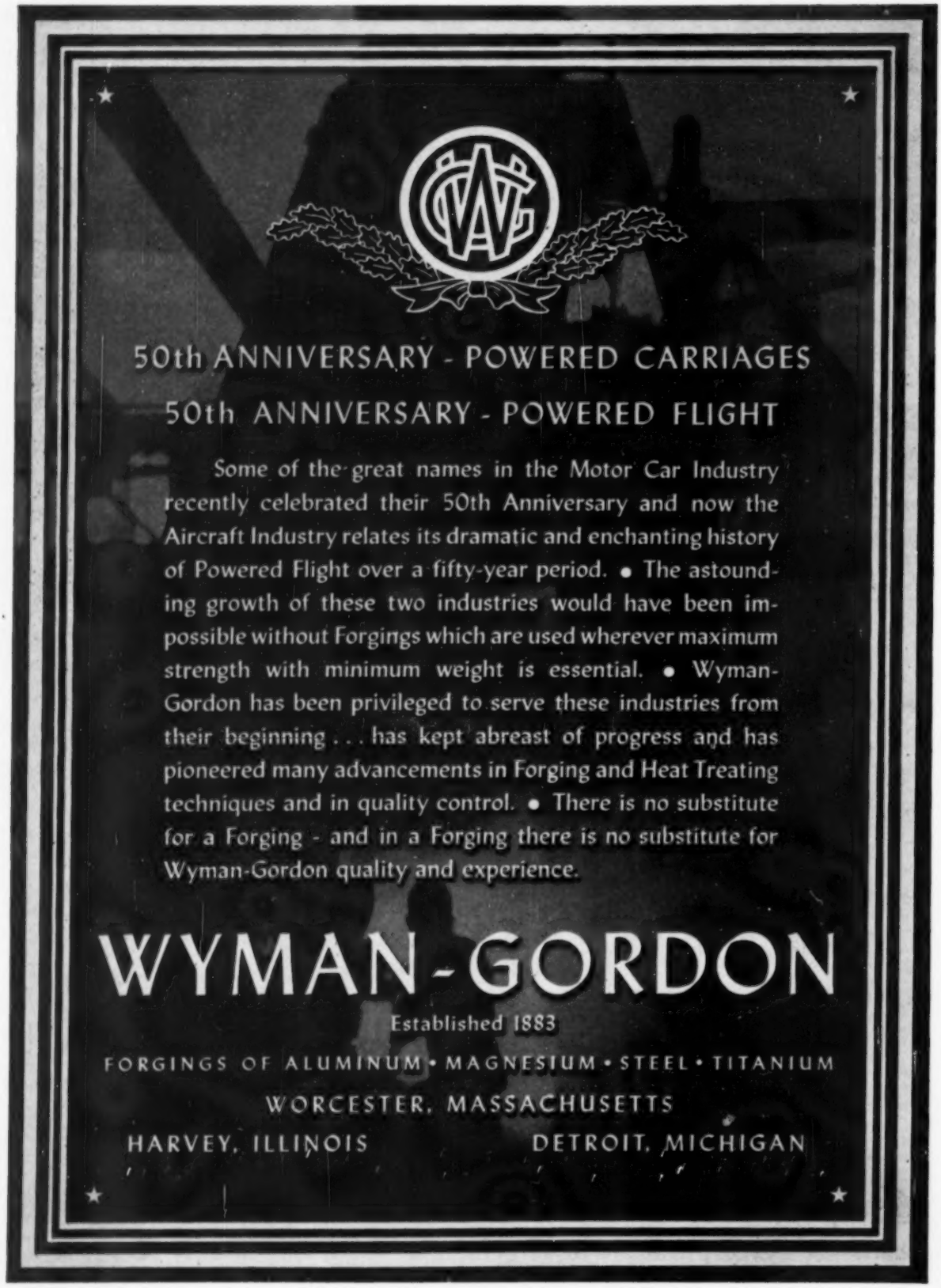
Wisconsin
Milwaukee—Zimmerman, Clyde R. (A)

Canada
Ottawa, Ontario—Teghtsoonian, Edward, Jr. (J)
Ottawa, Ontario—Weinberg, Fred (J)
Port Nelson, Ontario—McMulkin, Francis J. (R. C/S—S-M)
Toronto, Ontario—Reimers, Jan J. (M)

Italy
Genova—Bianchini, Osvaldo (M)

Japan
Koyama, Kamikyo-ku, Kyoto—Mori, Toshisada (A)
Nagamachi, Sendai—Sudo, Kingo (M)

South America
Chile, Chuquicamata—Moore, Robert C. (M) (R. C/S—J-M)



50th ANNIVERSARY - POWERED CARRIAGES

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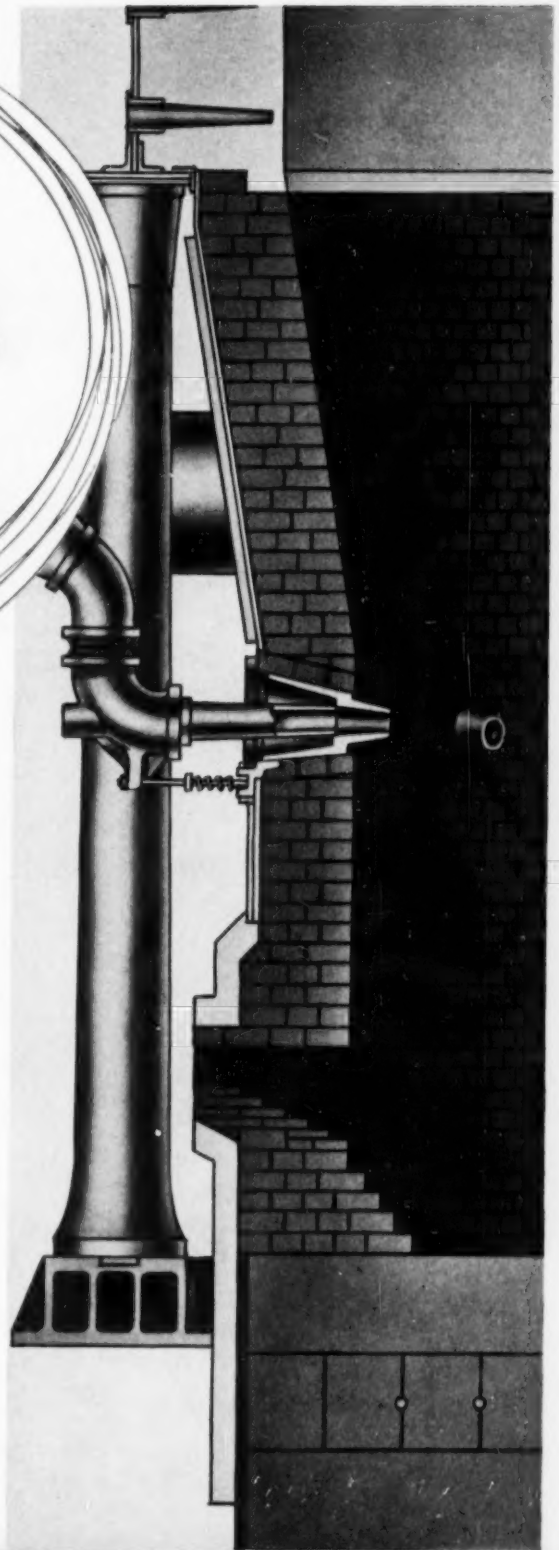

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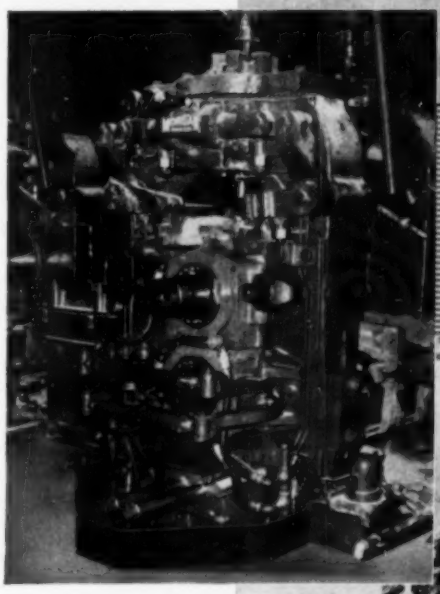
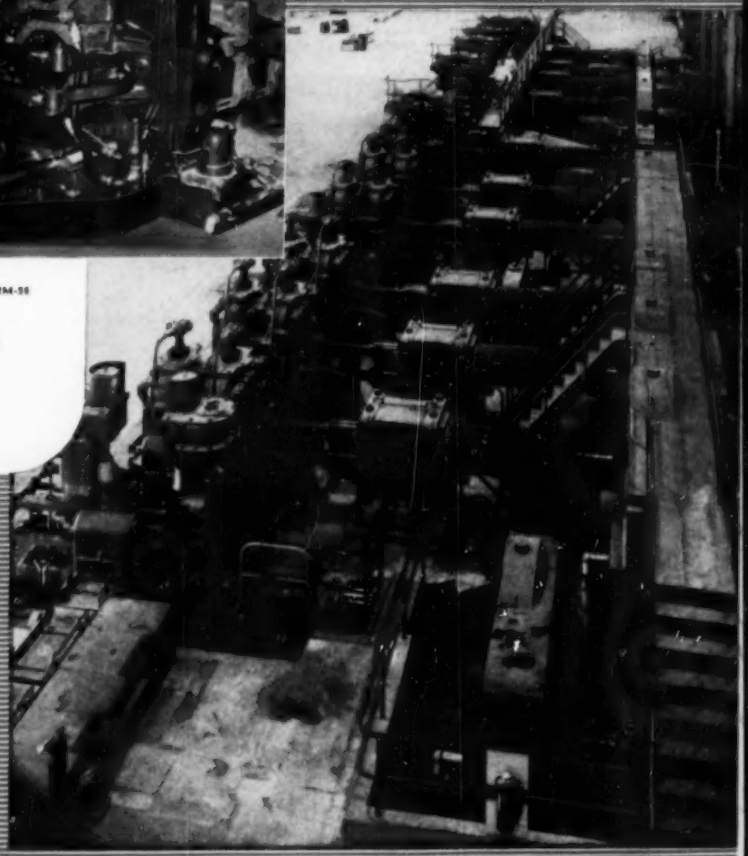
FAIRLESS Rolls Skelp with MORGAN Help

Hailed as one of the great strides toward the increase in American steelmaking capacity is the Fairless Works of the United States Steel Corporation at Morrisville, Pa. National Tube Division's 18" Skelp Mill, a vital part of this enterprise, was designed and built by Morgan Construction Company. Product gauge is assured by rigid closed top roll housings and MORGOIL roll neck bearings. Four ton slabs of steel become four ton coils of high quality skelp. For these, Morgan handling equipment meets exacting requirements for the safety and comfort of the mill operators with no loss of efficiency.





RM-55

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